

MID 064 197 742



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION 5  
77 WEST JACKSON BOULEVARD  
CHICAGO, IL 60604-3590

FEB 04 2016

REPLY TO THE ATTENTION OF:

**MEMORANDUM**

**SUBJECT:** EPA Data Request for Spatial Analysis via ArcGIS and Interactive Viewing in ePRISM.

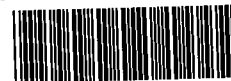
**FROM:** Juan Thomas, EPA Project Manager  
Conor Neal, EPA Hydrogeologist

**TO:** Michael Gerdenich, BASF Corporation

U.S. Environmental Protection Agency requests that BASF Northworks, through ARCADIS, set up an interactive spatial analysis tool in SharePoint. An ArcIMS (Arc Internet Map Service) database viewed in ePRISM will: allow for easier data sharing in the future, display the most current data available, allow for time- and spatial- trend analyses, expedite decision making, and considerably reduce reporting times moving forward. The geographic information system (GIS) should specifically contain the following information:

1. Base map of site
2. Shapefile of property boundary
3. Shapefile of most recent potentiometric surface contours
4. Shapefile outlining SWMUs and AOCs (figure 2)
5. Shapefile of current intermediate measures (IMs)
6. Shapefile of proposed capping locations (figure 20)
7. Shapefiles for each individually proposed corrective measure location, except those listed in request #5 above (figure 28);
  - a) Mixing zones,
  - b) Engineered discharge zones,
  - c) Funnel and gate, etc.
8. Shapefile for all locations where soil samples have been collected and hyperlinked in ePRISM to all available data for each sample location; data should be exportable for analysis
9. Shapefile for all locations where groundwater samples have been collected and hyperlinked in ePRISM to all available well data including boring log and quarterly sampling data; data should be exportable for analysis
10. Shapefile displaying isocontours for any constituent (metals, VOCs, SVOCs) that exceeds groundwater quality standards on-site (including, but not limited to, figures 3-10 and Appendix D, table 3)

US EPA RECORDS CENTER REGION 5



1004361

11. Shapefile displaying isocontours for any constituent (metals, VOCs, SVOCs) that exceeds soil quality standards on-site (including, but not limited to, figures 10-18)

\*Note: all figures and tables listed above are in reference to those found in the Draft Corrective Measures Study submitted to EPA June 25, 2015.

EPA requests that the ePRISM ArcIMS be operational in twenty-one (21) days from the date listed on this notice. We understand that all of the data and spatial information requested above is already available to BASF Northworks, as evidenced by the figures and tables in the Draft CMS.

The file paths for each figure indicate that BASF is already making use of ArcGIS for displaying data spatially. We believe that 21 days will suffice in the event of any technical difficulties that arise in the transfer of the data from desktop format to the ePRISM system. If you have any specific questions regarding the list above, please contact me at 312-886-6010 [thomas.juan@epa.gov](mailto:thomas.juan@epa.gov) or Conor Neal at (312) 886-7193, [neal.conor@epa.gov](mailto:neal.conor@epa.gov).

Sent via email



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77 WEST JACKSON BOULEVARD  
CHICAGO, IL 60604-3590

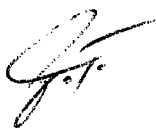
**FEB 04 2016**

REPLY TO THE ATTENTION OF:

**MEMORANDUM**

**DATE:** December 17, 2015  
(Resent February 4, 2016)

**SUBJECT:** CMS Overview Meeting of 12/15/2015 in Chicago

**FROM:** Juan Thomas, Corrective Action Project Manager 

**TO:** Michael Gerdenich, BASF Corporation

Mike, I just wanted to provide you a brief recap of some of the important points made at our meeting of 12/15, amongst the BASF Corporation Team, our EPA Team and the Team from MDEQ (on the conference line). Though we will also provide official comments to the draft CMS Report submitted by BASF, dated June 25, 2015, sometime early next 2016. I thought it important to document the items covered at the meeting that BASF can begin to address while awaiting additional comments from the CMS review.

EPA requests that BASF:

- conducts another soil gas sampling round in June 2016 (dry season) to account for temporal variability in soil gas movement.
- submits a revised final Tier 2 Risk Assessment addendum with the suggested changes such as ground water lead exposure evaluation, as well as an addition of a Human Health risk Assessment Conceptual Site Model that will include potential receptor contact pathways evaluation.
- considers risk management options for areas exceeding risk thresholds. e.g., AOC5 where HI = 2 due to redevelopment worker exposure to contaminated air during trench excavation.

EPA also requests, a supportive explanation as to how BASF proposes a Site-Wide Remediation Approach based upon SWMU/AOC Characterization Data. EPA understands that the site consists of a triangularly shaped wedge of fill 20/25+ feet thick along the River and feathering

out along the facility's western boundary. The fill consists/contains large amounts of chemical process wastes from the site's industrial past. Consequently, extrapolating from the SWMU/AOC characterization data to the proposed site-wide remedial approach should be discussed and justified. Similarly, the proposed cover areas (and more importantly, the non-cover areas) should account for not only the SWMU/AOC data but also the nature and heterogeneity of the fill. Additionally, the funnel & gate approach along the facility perimeter assumes the site's groundwater contamination is stable. BASF needs to provide an explanation and supporting data of plume stability.

EPA is not convinced that completion of the Legacy project prior to implementation of components of the RCRA Corrective Action for groundwater is appropriate. As discussed in the meeting, arguments supporting deferral of RCRA can be offset by comparable or better arguments being made for RCRA Corrective Action preceding the Legacy work. As such, it may be more appropriate to allow the two remedial efforts to proceed independently.

EPA would also like to have a GIS database set-up in order for BASF to share GIS data. Having the ability to visualize and spatially observe data might assist EPA in its evaluation process.

Also note that the CMS Report is to be stand-alone document. Providing a description of a remedial measure in a CMS report without providing a context of what the nature of the contamination that's being addressed as well as its extent is incomplete. The reader of the document should not have to refer to separate documents', i.e., the RFI Report and or Current Conditions Report to obtain a contextual understanding of what issues are being addressed with the selection of the proposed remedy. Providing some description as to what issues of concern are being addressed at the respective proposed remedial areas is required where relevant.

Lastly, the CMS Report presented the complete data tables from the southern boundary offsite investigation. The lab found matrix interference in many offsite samples, leading to extremely elevated detection limits above criteria in many locations. This was not previously conveyed to the Agencies, nor was the cause of this offsite contamination interference followed up on. The sampling design was based on quick turnaround time (24hr), an explanation of hold time exceedances in the analysis is warranted? Elevated pH was found at 13.9 in offsite groundwater, this is a direct contact hazard. The Agencies were not alerted to this data, its implications; nor has there been any follow-up on why this sample was elevated and what actions will be taken to address this immediate concern.

Sent via email



**From:** LARRY KYTE  
**To:** R5WST.R5RCRA.ORENSTEIN-BERNIE, R5WST.R5RCRA.NORLIN...  
**Date:** 4/30/99 10:59am  
**Subject:** ITS\_ENV False Claims Investigation by OIG

BASF  
NORTH  
WORKS

I am acting as a point of contact with the OIG which has requested certain information from the Region regarding samples analyzed by ITS-ENV where EPA funding was ultimately involved in payment for such services.

The Region has previously identified sampling at 6 facilities where that appears to be the case. Brian Freeman has acted as the contact for WPTD on the Region's ITS workgroup, and has coordinated the identification effort for the Division. Attached is a table that provides information regarding the sites and two other sites not paid for by EPA. All of those sites were apparently handled under the REPA contract within the past few years. Your group has been suggested to be the most likely depository of the specific documentary information requested by the OIG.

The OIG is looking for the following information regarding each of these 6 sites:

1. Site name
2. Amount paid for ITS-ENV data
3. Who the money was paid to (i.e, the name of the REPA contractor and then the subcontractor such as ITS-ENV or an intervening subcontractor)
4. The method of payment (contract such as REPA zone 2)
5. the number and type of samples required
6. the type of analysis required (EPA method)
7. A brief synopsis of the end use of the data and impact the data had on our decision making process, if any.
8. The current status or final disposition of the site.

Much of this information is known generally to us. Could you provide me with a brief synopsis of the information listed in 1-6 above by site? For example for Chevron site, XX Contractor contracted with ITS under REPA Zone 2 contract, XX soil samples and XX water samples, and XX sediment samples were collected, they were analyzed for XXX under Method 846. EPA was billed \$\$ for the collection of those samples and \$\$ for the analyses of those samples on XXX date. EPA paid those bills on xX date.

the I.G. will ultimately be wanting records that document the above facts and track the claim through the entire payment process. e.g., work statements, bills, payment vouchers, sample reports, etc.

**CC:** R5WST.R5RCRA.FREEMAN-BRIAN, R5WST.R5RCRA.SHARROW-D...

ITS Environmental Services Richardson, Texas

Site	Contractor	Sample Media	Action Taken
Chevron Oil R5 Attorney- Jerry Kujawa	TetraTech	RCRA sediment/soil samples	April 16, 1998 EPA letter to Chevron responds to Chevron's comment about ITS Richardson data problems, and leaves open issue of data use. ITS data corroborated by company data collected for corrective action.
Manistique Paper R5 Attorney- Deborah Garber	TechLaw	RCRA 17 groundwater and soil samples	Circa April 1, 1998 call -Respondent's attorney told of QA/QC problems; May 27, 1998 letter to Respondent's attorney confirms notice of ITS problem and need to resample in June. Resampling was conducted. ITS data will not be used.
USS Lead Refinery R5 attorney- Mike McClary	TechLaw	RCRA 7 groundwater and Soil samples	Corroborated by company data for corrective action. Will not rely on ITS data. A memo has been placed in Administrative record as to non-reliance on ITS-ENV data. A letter from the project manager, dated December 3, 1998, has been sent to the company notifying them of the ITS problem along with the memo to Administrative record..
BASF Wyandotte R5 Attorney- Reg Pallesen	TechLaw	RCRA 7 groundwater and soil samples	May 11, 1998 EPA letter notifying company of ITS problem. Split samples taken; ITS-ENV data corroborated by Company data.
WCI R5 Attorney- Diedre Tanaka	TechLaw	RCRA 1 soil sample	Sample data not being used in enforcement action. Complaint filed in March. Decision by Enforcement Team not to disclose until requested in Discovery. Documents relating to the ITS-ENV disclosure have been placed in files provided during discovery.

BASF Corporation

**BASF**

K. Edward Nuernberg  
General Manager  
Wyandotte Site

CERTIFIED MAIL – RETURN RECEIPT REQUESTED  
Z 058 363 542

March 3, 1999

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (DRE-9J)  
77 West Jackson Street  
Chicago, Illinois 60604

Subject: Submittal of RCRA Facility Investigation Report  
Docket No.: V-W-011-94  
BASF Corporation, Wyandotte, Michigan

Dear Ms. Sharrow:

BASF Corporation requested QST Environmental, the RFI Consultant, to send two copies of the RCRA Facility Investigation Report for the Wyandotte site directly to the EPA. You should receive the Report today.

This Report describes the results of the investigation at the North Work Site conducted during 1996 and 1997, and it is the basis for the CMS field program currently getting underway. The Report incorporates responses to the various sets of comments BASF Corporation received from the EPA.

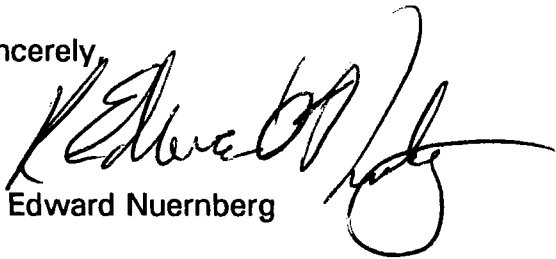
I certify that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to evaluate the information submitted. I certify that the information contained in or accompanying this submittal is true, accurate, and complete. As to those identified portion(s) of this submittal for which I cannot personally verify the accuracy, I certify that this submittal and all attachments were prepared in accordance with procedures designed to assure that qualified personnel properly gathered and evaluated the information submitted. Based on my inquiry of the person or persons who manage the system, or those directly responsible for gathering the information, or the immediate supervisor of such person(s), the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

March 3, 1999

Should you need additional information, please contact Mr. Jack Lanigan (734-324-6219) at your convenience.

Sincerely,

K. Edward Nuernberg

A handwritten signature in black ink, appearing to read 'K. Edward Nuernberg', with a large, stylized flourish at the end.

i:\ecology\jack\rfi-trans.doc

cc: J Lanigan - BASF  
MSutherland - Parsons  
RBlayer - MDEQ Lansing  
LAubuchon - MDEQ Livonia  
JRussell - MDEQ Livonia  
ADanford - Quanterra (letter only)  
BWallace - Bacon Memorial Public Library

CERTIFIED MAIL - RETURN RECEIPT REQUESTED  
P 607 933 868

November 23, 1998

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (DRE-9J)  
77 West Jackson Street  
Chicago, Illinois 60604

Subject: Revised Addendum to the ESI Data Validation Reports  
RCRA Facility Investigation  
Docket No.: V-W-011-94  
BASF Corporation, Wyandotte, Michigan

Dear Ms. Sharrow:

BASF Corporation is submitting two copies of the Revised Addendum to the Data Validation Reports for the BASF Northworks Facility RFI in Wyandotte, Michigan (including cover letter). The revised addendum was received from Environmental Standards Inc. on November 19.

As I mentioned during our telephone conversation on November 20, errors were discovered in four, ESI addendum reports that had been submitted to BASF. ESI chose to reissue the entire addendum; therefore, please destroy your copies of the earlier addendum. ESI's letter dated November 4 is still valid and should be retained.

As I stated earlier, I will revise the consolidated validated data tables and send a copy under separate cover to MDEQ and to the Bacon Memorial Public Library in Wyandotte.

Should you need additional information, please contact us at your convenience.

Sincerely yours,



Bruce Roberts  
Project Manager

Attachment - Revised Data Validation Addendum and ESI cover letter

cc: E. Nuernberg - BASF  
J. Byrnes - BASF  
D. Marian - QST  
R. Blayer - MDEQ Lansing (letters only)  
L. Aubuchon - MDEQ Livonia (letters only)  
J. Russell - MDEQ Livonia (letters only)  
A. Danford - Quanterra (letters only)  
B. Wallace - Bacon Memorial Public Library (letters only)



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5  
77 WEST JACKSON BOULEVARD  
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF  
DE-9J

COPY

30 September 1998

Bruce Roberts, Project Manager  
BASF Corporation  
1609 Biddle Avenue  
Wyandotte, Michigan 48192

RE: BASF North Works, Wyandotte, Michigan  
U.S. EPA Identification Number MID 064 197 742  
Draft Response to EPA's Data validation Comments

Dear Mr. Roberts:

The United States Environmental Protection Agency (U.S.EPA), Region 5, has received and reviewed your letter of September 15, 1998, including the attachments. U.S. EPA accepts all of the proposed responses and edits as outlined in Environmental Standards, Incorporated's (ESE), letter of September 11, 1998. Accordingly, please submit the revised RFI report.

In response to ESE's request for feedback; there were no other specific data validation reports, other than those noted, in which the application of the blank qualification or data qualification was questioned.

If you have any questions, please contact me at (312) 886-6199.

Sincerely,

A handwritten signature in dark ink, appearing to read "DMS".

Diane M. Sharrow  
Project Manager  
U.S. EPA, Region 5  
Waste, Pesticides and Toxics Division  
Enforcement and Compliance Assurance Branch

cc: Reginald Pallesen, ORC



Setting the Standards for Innovative  
Environmental Solutions

M W Regional Ecology

SEP 14 1998

September 11, 1998

Mr. Bruce D. Roberts  
Project Manager  
BASF Corporation, Inc.  
1609 Biddle Avenue  
Wyandotte, MI 48192

**Subject: Response to United States Environmental Protection Agency Region 5's Review of the Data Validation and Usability of Data for the Draft RFI Report for the BASF Northworks Facility in Wyandotte, Michigan.**

Dear Mr. Roberts:

Environmental Standards, Inc. (Environmental Standards) has received a copy of the letter dated May 11, 1998, from the United States Environmental Protection Agency (U.S. EPA) Region 5 addressed to you concerning the issues raised in the review of the data validation and usability of data for the BASF Northworks Facility in Wyandotte, Michigan. Environmental Standards would like to thank U.S. EPA Region 5 for the compliment of stating that the validation effort appeared to be comprehensive and appropriately conservative. U.S. EPA Region 5 included comments and observations indicating that the data was thoroughly evaluated. Environmental Standards takes pride in the work performed and is viewed as a leader in the industry for third-party data validation.

In order to effectively address the questions and requested clarifications raised by U.S. EPA Region 5, Environmental Standards has outlined the complete May 11, 1998, U.S. EPA Region 5 letter. Environmental Standards' proposed responses, edits, and requests for further clarifications are presented in *italics* following each issue raised by U.S. EPA Region 5. If the proposed responses, clarifications, and edits provided herein are acceptable to the U.S. EPA Region 5, an addendum to the applicable previously issued data validation reports will be provided.

**ENVIRONMENTAL STANDARDS, INC.**  
VALLEY FORGE, PA

1140 Valley Forge Road, P.O. Box 911, Valley Forge, PA 19482-0911 • 610-935-5577 • Internet [OffNPL@EnvStd.com](mailto:OffNPL@EnvStd.com)  
1111 Kennedy Place, Suite 2, Davis, CA 95616 • 916-758-1903 • Internet [ENVSTDWEST@AOL.com](mailto:ENVSTDWEST@AOL.com)  
Copper Bend Centre, 956 South 59th Street, Belleville, IL 62223 • 618-257-3800 • Internet [MIDWEST@EnvStd.com](mailto:MIDWEST@EnvStd.com)  
World Wide Web [HTTP://www.EnvStd.com/](http://www.EnvStd.com/)

U.S. EPA Region 5 General Comments:

1. "In several of the Data Validation Reports, sample results qualified as non-detected and flagged "U" were not further qualified as estimates and flagged "J" for QC deficiencies which required the qualification of non-detects. Examples include, but are not limited to, the following.
  - In Data Validation Report 5, *bis*(2-ethylhexyl)phthalate was qualified "U" due to blank contamination in samples SG005SWMUFSP07, SG010SWMUFSP06 and SG011SWMUFSP18. However, it appears that the results should have been qualified "UJ" to also account for low internal standard areas.
  - In Data Validation Report 7, antimony in sample SG002AOC6SP55 should be qualified "J" due to matrix spike recoveries, in addition to the "U" qualifier applied as a result of blank contamination.
  - In Data Validation Report 9, the antimony results for all samples should be qualified "J" due to matrix recoveries, in addition to the "U" qualifier applied due to blank contamination.
  - In Data Validation Report 10, methylene chloride should be qualified "J" in samples SG008AOC23 and SG001SWMUG8 due to low surrogate recoveries, in addition to the "U" qualifier applied as a result of blank contamination.

BASF should revise the validation reports and all associated sections and tables of the RFI to reflect all changes in the qualifiers assigned in addressing this issue. Alternatively, BASF should provide the rationale for not assigning those qualifiers associated with non-detects to results flagged "U" due to blank contamination."

*Data qualified as "U\*" as a result of blank contamination was not further qualified due to additional QA/QC limitations because blank qualification of data takes precedence over any additional data qualification. There is a hierarchy of data qualification codes that Environmental Standards employs. Blank qualified data ("U\*") takes precedence over rejected data ("R") which takes precedence over estimated data ("J/UJ").*

*The qualification of analytical data as "U\*" due to blank contamination indicates that the laboratory-reported positive result cannot be definitively attributed to the investigative sample or to introduced contamination. As such, further qualification of data that has already been qualified due to blank contamination appears superfluous.*





*For this reason, the use of multiple data qualification flags is never applied by Environmental Standards.*

*Please note that for data qualifiers, the additional reasons for qualifying the analytical data are alluded to through the phrase "(unless previously flagged "U\*")." This parenthetical phrase refers to additional reasons for qualification if the data had not already been qualified due to blank contamination.*

2. "It appears that the data reviewers were not consistent in the reporting of sample results on the Analytical Results tables for those results greater than the laboratory reporting limits and qualified as non-detects (flagged "U") due to blank contamination. According to the *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (Functional Guidelines)*, the reporting limit should be replaced by the detected concentration and qualified "U". For example, if a compound with a reported result of 10 ug/kg and a reporting limit of 5 ug/kg were qualified "U" due to blank contamination, the validated results should be 10 U, not 5 U. Revise the data validation as necessary to address this issue, ensuring that all affected validation reports, analytical summaries and Data Qualifier discussions are appropriately revised. If professional judgment was used to take an action other than that specified in the *Functional Guidelines*, provide a thorough rationale for this decision."

*Of the 36 data validation reports issued by Environmental Standards, 13 had organic data that were qualified due to blank contamination. Of these 13 data validation reports, three were observed to have inconsistencies with the reporting of blank qualification on the associated data tables. These three data validation reports include: Report 10, Report 33, and Report 34. The data tables associated with these three reports will be revised to be consistent with the data flagging convention as described above. For example, in Report 10, the positive result for methylene chloride in sample SS001 SWMUG-6 will be qualified on the associated data tables as:*

Compound	Result (µg/kg)	Detection Limit	Final Qualifier
methylene chloride	6.6	5.5	U*

*Environmental Standards would appreciate feed-back from U.S. EPA Region 5 if there were any specific data validation reports (other than the three listed above) in which the application of blank qualification was questioned.*



3. "In those cases where samples were reanalyzed for organic parameters, the validation reports provide no indication of which set of results are of better quality and should, therefore, be used. In addition, in many cases the reanalyses were not clearly identified in the Analytical Results tables. Examples include, but are far from limited to:
- In Data Validation Report 1, the results for sample SG001RFIMW24 and its reanalysis were validated and reported. It appears that the reanalysis may be the better of the two analytical runs, since all non-detects in the original acid fraction were rejected, but all results were qualified only as estimates (due to exceeded holding time) in the reanalysis.
  - In Data Validation Report 2, the results for sample SG013RFIMW07 and its reanalysis are reported. However, no reason is given for the reanalysis and, therefore, no determination as to which sample results to be used can be made.
  - In Data Validation Report 5, the results for sample SG006SWMUFP11 and its reanalysis are reported. However, both samples exhibited the same QC criteria out of control, most likely indicating that the original analysis should be reported and used for decision-making purposes.

In each case where a sample was reanalyzed, revise all appropriate sections and/or appendices of the RFI Report to clearly indicate which analysis appears to be of better quality, based on the validation criteria, and to clearly indicate which set of results is reported and to be used for decision-making purposes."

*In those cases where samples were reanalyzed for organic parameters, Environmental Standards was not involved in the decision making process related to the selection of which set of results were of better quality. It is understood that BASF has supplied U.S. EPA Region 5 with the decision tree that was used by BASF in choosing the "best of" results.*

4. "Filtered and unfiltered inorganic results and field duplicate results were qualified based on poor agreement between the two sets of results for a given sampling location. Since validation based on the agreement between filtered and unfiltered results or field duplicate results are not addressed in the *USEPA Contract Laboratory Program*



*National Functional Guidelines for Inorganic Data Review*, it appears that professional judgment was used to apply the qualifiers. Furthermore, it may be that either an unnecessarily conservative criterion was used, or errors exist in the application of qualifiers due to this issue. See for example the barium results for sample MW000RFIMW3 in Data Validation Report 25. Provide the rationale and criteria used to make all such judgments."

*Environmental Standards followed the guidelines established in the project specific data validation SOPs. The criteria for the filtered/unfiltered data qualification was developed with guidance from U.S. EPA Region 2 specifications, as this is the only regulatory agency that provides guidelines for this circumstance. Additionally, the filtered/unfiltered data evaluation forms provided in the data validation report's support documentation provides the criteria used to assess data quality as well as any data qualification resulting from the filtered/unfiltered data evaluation process.*

*In the data validation SOPs, the following criteria were used for the evaluation of filtered and unfiltered results:*

*If one of both of the results (total and filtered concentrations) are less than 10× the reporting limit (RL), use the following criteria:*

- a. If the difference between the results is greater than the RL, flag both results as estimated ("J").*
- b. If the difference between the results is greater than 5× RL, flag both results as unreliable ("R").*

*If both results are greater than 10× RL, then calculate the percent difference using the following equation:*

$$\%D = \frac{\text{Total Concentration} - \text{Filtered Concentration}}{\text{Total Concentration}}$$



*Apply the following criteria:*

- a. *If the percent difference is greater than 10%, flag both results as estimated ("J").*
- b. *If the percent difference is greater than 50%, flag both results as unreliable ("R").*

*The abovementioned example of barium results of sample MW000RFIMW3 in Data Validation Report 25 falls into the scenario where both results are greater than  $10 \times RL$  and the percent difference between the two positive results are greater than 10%. Under the data validation guidelines, both positive results (i.e. total and filtered barium) in sample MW000RFIMW3 should be flagged as estimated ("J").*

*The following criteria were established for field duplicate results:*

- a. *If the RPD is  $> 20\%$  for aqueous samples or  $40\%$  for solid samples (when sample and duplicate results were  $\geq 5 \times RL$ ), flag all associated positive results "J."*
- b. *If the control limit of  $\pm RL$  for aqueous samples or  $2 \times RL$  for soil samples for results  $< 5 \times RL$  (either sample or duplicate) is exceeded, flag all associated positive results "J." If a results is less than the RL, use the RL for comparison purposes.*

*The field duplicate criteria were developed using professional judgment. Additionally, the field duplicate data evaluation forms provided in the data validation report's support documentation provides the criteria used to assess data quality as well as any data qualification resulting from the field duplicate evaluation process.*

*Environmental Standards would appreciate feed back from U.S. EPA Region 5 if there were any specific data validation reports in which the application of data qualification regarding filtered and unfiltered inorganic results and/or field duplicate results were questioned.*



SPECIFIC COMMENTS:

1. "Report 1 (SDG A6G1701300)- The Analytical Results tables for the inorganic analyses could not be located in Section 2, Analytical Results. Based on the text, it appears that validation was appropriately performed, however, no verification of the assignment of qualifiers could be made. Provide the missing tables."

*An addendum of the missing tables will be provided.*

2. "Report 2 (SDG A6G250145)- According to Item 11 of the Organic Data Qualifiers in Section 1, the volatile organic compounds (VOCs) quantitated using the internal standard chlorobenzene-d5 were qualified in sample SG004RFIMW07. However, the qualifiers were not included on the Analytical Results table on page 1 and 2 of Section 2. Revise the Data Validation Report to address this discrepancy."

*The corresponding Analytical Results page(s) of the data validation report will be corrected and reissued with the corresponding VOCs qualified in sample SG004RFIMW07.*

3. "Report 4 (SDG A6310114) - Item 1 of the Organic Data Qualifiers in Section 1 incorrectly indicates that pentachlorobenzene has been rejected. As accurately reflected in the Analytical Results table in Section 2, the compound for which the result was rejected is actually pentachloronitrobenzene. Revise the Data Validation Report to address this discrepancy."

*The corresponding page(s) of the data validation report will be corrected and reissued.*

4. "Report 5 (SDG A6H020118) - For sample SG010SWMUFSP06, the compound chloroethane, rather than chlorodibromomethane, should have the "UJ" qualifier on page 10 of the Volatile Analytical Results table in Section 2. Revise the Data Validation Report to address this transcription error."

*The corresponding Analytical Results page(s) of the data validation report will be corrected and reissued.*

5. "Report 5 (SDG A6H020118) - For sample SG006SWMUFSP11, the compound pentachloronitrobenzene, rather than pentachlorophenol, should have the "UR"



qualifier on page 19 of the Semivolatile Analytical Results table in Section 2. Revise the Data Validation Report to address this transcription error."

*The corresponding Analytical Results page(s) of the data validation report will be corrected and reissued.*

6. "Report 6 (SDG A6H030119) - The pH results are not included in the Analytical Results tables in Section 2. Revise the tables to include the pH results."

*The corresponding Analytical Results page(s) of the data validation report will be corrected and reissued.*

7. "Report 7 (SDG A6H150150) - Sample RS000AOC6 was incorrectly identified as AS000AOC6 in Item 2 of the Organic Data Qualifiers section. Correct this typographical error."

*The corresponding page(s) of the data validation report will be corrected and reissued.*

8. "Report 8 (SDG A6H160167)- The Data Validation Report does not address the low recoveries for surrogate TCX and the zero percent recoveries for DCB reported on Form 2 for sample SD004SWMUE4 in the pesticide/PCB results. It appears that all non-detected results for this sample should be rejected. Either provide a thorough rationale for not rejecting the results, or revise as necessary all affected sections of the Data Validation Report and the text of the RFI Report to address the rejection of these results."

*The pesticide/PCB results reported for sample SD004SWMUE4 were reported from the primary column (RTX1701). The recovery of TCX from this column was 20% (within the acceptance limits of 14-155%); however, the recovery of DCB from this column was 0%. Therefore, all non-detected pesticide/PCB results for sample SD004SWMUE4 should have been rejected and flagged "UR" on the associated data tables. Please note that there were no positive results associated with the pesticide/PCB analysis of sample SD004SWMUE4, and therefore, only "UR" flags will be applied. The appropriate section of the data validation report and the associated pesticide/PCB Analytical Results table will be revised to reflect this situation.*

9. "Report 8 (SDG A6H160167) - Item 2 of the Organic Data Qualifiers section identifies an incorrect sample number. The actual sample qualified appears to be



SD003SWMUE1, rather than SD004SWMUE4, as reflected in the Analytical Results tables. Revise the Data Validation Report to address this discrepancy.”

*The corresponding page(s) of the data validation report will be corrected and reissued.*

10. “Report 8 (SDG A6H160167) - According to the Volatiles Analytical Results tables, 2-butanone (MEK) was qualified as “UJ” in samples SD003SWMUE2 and SD004SWMUE4. However, the Organic Data Qualifiers section does not address this compound. Revise the Data Validation Report to address this inconsistency.”

*2-Butanone (MEK) should not have been flagged as “UJ” in samples SD003SWMUE2 and SD004SWMUE4. The “UJ” flags will be removed from the associated Volatiles Analytical Results tables and the corresponding pages of the Analytical Results tables will be reissued.*

11. “Report 9 (SDG A6H160149) - 1,2-Dichloroethane was rejected and flagged “R” in samples SG002AOC6SP63 and SG002AOC6SP64 according to the Volatile Analytical Results tables. However, the Organic Data Qualifiers section provides no reason for these qualifiers. Revise the Data Validation Report to address this issue.”

*1,2-Dichloroethane was inadvertently rejected and flagged “UR” in samples SG002AOC6SP63 and SG002AOC6SP64 due to the low internal standard recovery (<25%) associated with chlorobenzene-d5. 1,2-Dichloroethane is not quantitated from the response associated with chlorobenzene-d5 and therefore should not have been flagged “UR” on the associated data tables. The corresponding Volatile Analytical Results tables will be modified to reflect this situation.*

12. “Report 10 (SDG A6H220161) - Several VOCs were apparently incorrectly qualified for low internal standard areas based on a review of relative retention times found on page 378 of the associated data package. Tetrachloroethene, toluene and 4-methyl-2-pentanone should also be qualified “UJ” for those samples qualified due to low chlorobenzene-d5 areas. In addition, EDB and chlorodibromomethane are not associated with chlorobenzene-d5, but with internal standard 1,4-difluorobenzene and should not, therefore, be qualified in those samples with chlorobenzene-d5 as the only internal standard with areas outside the QC limits. Revise the Data Validation Report to address this issue.”



*The corresponding Analytical Results page(s) of the data validation report will be corrected and reissued.*

13. "Report 10 (SDG A6H220161) - The compound phenacetin was qualified "UJ" in the Semivolatile Analytical Results tables for sample SS001SWMUG4. However, the Organic Data Qualifiers section does not address this qualifier. Revise the Data Validation Report to correct this discrepancy."

*Phenacetin in the Semivolatile Analytical Results tables for sample SS001SWMUG4 should not have been qualified "UJ". The corresponding page(s) of the Semivolatile Analytical Results tables will be corrected and reissued.*

14. "Report 10 (SDG A6H220161) - It is indicated under the Noncorrectable Deficiency, Item 4, listed on page 4 of Section 1, that the impact of the deficiency is discussed in the Organic Qualifier Section. However, said discussion could not be found, nor were qualifiers applied to the associated compound, methoxychlor. Revise the Data Validation Report to provide this information and any associated qualifiers."

*The analytical data associated with methoxychlor was not impacted due to this deficiency. Item 4 of the Noncorrectable Deficiency section will be revised to reflect this situation.*

15. "Report 11 (SDG A6H240102) - It is indicated in the Inorganic Data Qualifiers section that the zinc result for sample SG001AOC26 should be qualified "J". Revise the Analytical Results table to include this qualifier."

*The corresponding Analytical Results page(s) of the data validation report will be corrected and reissued.*

16. "Report 12 (SDG A6H240106) - It appears that the zinc result for sample RS00AOC7, an aqueous sample, was incorrectly qualified based on the results of the soil ICP serial dilution. Revise the Data Validation Report as necessary to address this issue."

*The corresponding page(s) of the data validation report will be corrected and reissued.*

17. "Report 13 (SDG A6H270124) - All results for sample RS000AOC2 were qualified "UJ/J" because the sample was analyzed one day beyond the seven-day holding time. According to the *Functional Guidelines*, only aromatic compounds should be qualified





in unpreserved aqueous samples when the seven-day holding time is exceeded. All other compounds have a 14-day holding time. (The Data Validation Report does not clearly indicate that the sample was unpreserved; it is only so inferred based on the application of the qualifiers). Revise the Data Validation Report as necessary to address this apparent unnecessarily conservative application of qualifiers."

*Only the aromatic compounds should have been qualified in sample RS000AOC2. The corresponding page(s) of the data validation report and the Volatile Analytical Results tables will be corrected and reissued.*

18. "Report 15 (SDG A6I100127) - It is indicated in the Organic Data Qualifiers section that the 1,3,5-trinitrobenzene result for sample SW000AOC7 should be qualified "UR". Revise the Semivolatile Analytical Results table to include this qualifier."

*The corresponding Analytical Results page(s) of the data validation report will be corrected and reissued.*

19. "Report 24 (SDG A6I250139) - According to Item 4 of the Organic Data Qualifiers section, the 4-nitrophenol results should be qualified in two of the four reported samples. However, the results for this compound are qualified in all four samples on the Semivolatile Analytical Results tables. Revise either the text or table as necessary to address this discrepancy."

*The non-detected results for 4-nitrophenol only in samples MW000RFEMW-6 and MW000RFEMW-7 should have been flagged "UJ" due to high percent drifts (> 25%) in the associated continuing calibration standards. The "UJ" flags associated with samples MW000RFEMW-4 and MW000RFEMW-5 will be removed from the corresponding Semivolatile Analytical Results tables.*

20. "Report 30 (SDG A6J010134) - The second item of the Noncorrectable Deficiencies section indicates that the holding times were exceeded by 20 days for samples MW000RFIMW-29, MW000RFIMW30 and TB-10/1/96. For these samples, the holding times have been grossly exceeded and all VOC non-detect results should be rejected and flagged "R". Either revise all associated sections and tables of the Data Validation Report and main RFI Report to address these rejections, or provide a thorough rationale for deviating from the *Functional Guidelines*."



*For samples MW000RFIMW-29, MW000RFIMW30 and TB-10/1/96, the holding times have been grossly exceeded, and all VOC non-detect results should be rejected and flagged "UR." The corresponding page(s) of the data validation report and the Volatile Analytical Results tables will be corrected and reissued.*

21. "Report 30 (SDG A6J010134) - The second item in the Inorganic and Wet Chemistry Data Qualifiers section indicates that the cyanide samples were of a soil matrix. However, the Analyte Results Summary and supporting documents indicate a water matrix. Revise the Data Validation Report to address this discrepancy."

*The second item in the Inorganic and Wet Chemistry Data Qualifiers section incorrectly states that the cyanide samples MW000RFIMW-29 and MW000RFIMW30 were of a soil matrix. The Analyte Results Summary and supporting documents correctly indicate the matrix as water. The second item in the Inorganic and Wet Chemistry Data Qualifiers section on page 14 of the data validation report will be changed to indicate an aqueous matrix.*

22. "Report 32 (SDG A6K200114) - All VOC results (in sample MW000RFIMW-28), except those previously flagged "UR", were qualified as estimated due to exceeded holding times. However, it is indicated in Item 5 of the Organic Data Qualifiers section that only the aromatic compounds were qualified. Revise the Data Validation Report to resolve this discrepancy."

*As indicated in Item 5 of the Organic Data Qualifiers section of the data validation report, only the aromatic compounds should have been qualified in sample MW000RFIMW-28. The corresponding page(s) of the Volatile Analytical Results tables will be corrected and reissued.*

23. "Report 33 (SDG A6L190128) - The analytical results table for the VOC analysis of sample MW000RFIMW9 was not included in Section 2, Analytical Results. While the reported validation appears to be accurate, the application of qualifiers could not be confirmed. Revise the Data Validation Report to include this table."

*The corresponding Analytical Results page(s) of the data validation report will be reissued.*

24. "Report 33 (SDG A6L190128) - Several results were rejected and flagged "R" due to very low Laboratory Control Sample (LCS) recoveries. However, validation due to



LCS results are typically based on project-specific criteria. No such criteria are presented. Revise the Data Validation Report to discuss the criteria used to qualify results based on LCS recoveries."

*Report 33 states the following:*

*"The actual quantitation limits for 2-chlorophenol in samples MW00ORFIMW-10 (MS/MSD) RE-1, MW00OPMINA, MW00ORFIMW-29, MW00ORFIMW-27, MW00OP-34-N, MW00ORFIMW-25, MW00ORFIMW-25D, MW00ORFIMW-24 (MS/MSD), MW00ORFIMW-26, and RS00ORFIMW-26 and for pentachlorophenol, phenol, and 4-chloro-3-methylphenol in sample MW00ORFIMW-10 (MS/MSD) RE-1 are unusable, and the "not-detected" results have been flagged "UR" on the data tables. Low recoveries were observed for these compounds in the associated LCS analyses."*

*The laboratory reported recovery windows were the criteria that was used in the assessment of the associated aqueous LCS analyses. In the abovementioned situation, the recoveries of the compounds were less than the lower limit and, therefore all associated "not-detected" results should have been flagged "UJ" instead of "UR" on the data tables. The corresponding page(s) of the data validation report and the Analytical Results tables will be corrected and reissued.*

25. "Report 34 (SDG A7C190143) - Several semivolatile results were rejected and flagged "R" due to very low Laboratory Control Sample (LCS) recoveries. However, validation due to LCS results are typically based on project-specific criteria. No such criteria are presented. Revise the Data Validation Report to discuss the criteria used to qualify results based on LCS recoveries."

*Report 34 states the following:*

*The actual quantitation limits for 1,2,4-trichlorobenzene, acenaphthene, 2,4-dinitrotoluene, pyrene, and 4-chloro-3-methylphenol in samples MW00ORFIMW-25, MW00ORFIMW-26, MW00ORFIMW-27, MW00OP-34-N, MW00ORFIMW-28, MW00ORFIMW-22, MW00ORFIMW-24, MW00ORFIMW-3, MW00ORFIMW-4, MW00ORFIMW-23, MW00ORFIMW-2, MW00ORFIMW-10, MW00ORFIMW-5, MW00ORFIMW-6, MW00ORFIMW-7, MW00ORFIMW-30, MW00ORFIMW-12, MW00ORFIMW-11, MW00ORFIMW-29, and MW00OMWPMINA may be higher than reported and the "not-detected" results have been flagged "UJ" (unless previously flagged*



"UR") on the data tables. The reported positive results for pyrene in sample MW000RFIMW-23; for acenaphthene in samples MW000RFIMW-7 and MW000RFIMW-30 should be considered estimated and have been flagged "J" on the data tables. Low recoveries were observed for these compounds in the associated laboratory control sample analysis.

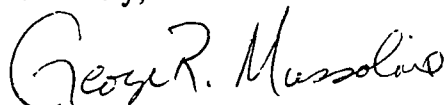
The laboratory reported recovery windows were the criteria that was used in the assessment of the associated aqueous LCS analyses. In the abovementioned situation, the recoveries of the compounds were less than the lower limit and, therefore all associated "not-detected" results should have been flagged "UJ" on the data tables. No corrections to the data validation report or the Analytical Results tables are necessary.

26. "Report 35 (SDG A7F030144)" - It appears that Item 7 in the Inorganic Data Qualifier section incorrectly identified the qualifier due to RPD criteria as "U\*". The qualifier should be "UJ" unless previously qualified due to blank contamination. Revise the Data Validation Report to address this apparent discrepancy."

*The corresponding Analytical Results page(s) of the data validation report will be reissued.*


If you have any questions or comments, or if I can be of any further assistance, please feel free to call.

Sincerely,



George R. Mussoline  
Senior Quality Assurance Chemist II/  
Project Manager

Concurred by:



Rock J. Vitale, CPC  
Director of Chemistry





UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5  
77 WEST JACKSON BOULEVARD  
CHICAGO, IL 60604-3590

COPY

REPLY TO THE ATTENTION OF:  
DE-9J

11 May 1998

Thomas F. McGourty, Manager  
Ecology Services Department  
BASF Corporation, Incorporated  
1609 Biddle Avenue  
Wyandotte, Michigan 48192

RE: BASF North Works, Wyandotte, Michigan  
U.S. EPA Identification Number MID 064 197 742  
Land Reclamation Projects

Dear Mr. McGourty:

The United States Environmental Protection Agency (U.S. EPA), Region 5, has received your letter of March 27, 1998, regarding the Land Reclamation Projects (Projects), proposed for the Tar Pit at AOC 4 and the Prussian Blue at AOC 7C.

As we discussed during our meeting on March 16, 1998, the Projects do not seem to fit the Interim Measures / Stabilization Criteria under the Administrative Consent Order (Order), Docket V-W-94-R-011. However, the Projects do involve AOCs included in the Order.

After review of the Projects and the Order, U.S. EPA has determined that, although the proposed Projects are consistent with the mutual objectives of BASF Corporation, Inc., (BASF) and U.S. EPA, as outlined in Section IV of the Order, that:

- 1) the proposed Projects cannot be considered Interim Measures under the terms of the Order, because there are no emergency conditions to be addressed; and
- 2) the proposed Projects cannot be determined to be Corrective Measures under the terms of the Order, because the projects are in advance of a Corrective Measures Study (CMS).

Nonetheless, BASF has presented compelling reasons for timely implementation of the proposed Projects; i.e., it is highly unlikely that implementation of the proposed Projects will cause, contribute to, or exacerbate the release, or potential release, of hazardous wastes or hazardous constituents; and, if

successfully implemented, the proposed Projects appear likely to be approved as Corrective Measures.

Accordingly, U.S. EPA will not prohibit the timely implementation of the proposed Projects, provided that BASF complies with the following conditions and requirements:

- 1) that BASF submit to U.S. EPA oversight of the proposed Projects, as set out below;
- 2) that implementation of the proposed Projects not delay submission of the CMS required by the Order;
- 3) that BASF assure the opportunity for public participation regarding the proposed Projects by providing notice and information about the implementation, progress and results of the proposed Projects through the established Community Advisory Panel; and
- 4) that BASF include in the CMS required by the Order, all relevant information about the implementation, progress and results of the proposed Projects, which is available at the time of the CMS submittal.

The U.S. EPA oversight of the proposed Projects shall consist of the following:

- 1) advance notice to U.S. EPA by BASF of any construction or remediation/removal activities, including the selection of any full-scale action at AOC 4;
- 2) advance authorization by U.S. EPA prior to construction and remediation/removal activities; and
- 3) the submittal to U.S. EPA, by the tenth of each month, of a written Monthly Report which summarizes details about each of the Projects.

Each Monthly Report shall address key items such as those normally included in a Corrective Measures Work plan and Report. U.S. EPA expects that the compiled Monthly Reports will provide a basis for both including the proposed Projects in the CMS and, upon completion of the Projects, preparing a Final Report on each of the Projects. U.S. EPA will comment on the Monthly Reports as necessary.

U.S. EPA expects that BASF will notify the Agency by telephone, or in writing, of any major alterations, problems or emergency occurrences regarding the Projects, and not wait until the Monthly Report is submitted. U.S. EPA's agreement to allow BASF to proceed with the proposed Projects does not constitute an advance approval of the Projects as Corrective Measures under the Order.

Thank you for your discussions and letter. If you have any questions regarding U.S. EPA's response, please contact me at (312) 886-6199.

Sincerely,

Diane M. Sharrow, Project Manager  
U.S. EPA, Region 5  
Waste, Pesticides and Toxics Division  
Enforcement and Compliance Assurance Branch

cc: Bruce Roberts, BASF

bcc: Reginald Pallesen, ORC

bcc: Reginald Pallesen, ORC





UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5  
77 WEST JACKSON BOULEVARD  
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

DE-9J

11 May 1998

COPY

Bruce D. Roberts, Project Manager  
BASF Corporation, Incorporated  
1609 Biddle Avenue  
Wyandotte, Michigan 48192

RE: BASF North Works, Wyandotte, Michigan  
U.S. EPA Identification Number MID 064 197 742  
Draft Phase I RCRA Facility Investigation Report

Dear Mr. Roberts:

The United States Environmental Protection Agency (U.S.EPA), Region 5, has received and reviewed the BASF Corporation, Incorporated, (BASF), Draft Phase I RCRA Facility Investigation Report (RFI), of December 4, 1997. The U.S. EPA is approving the RFI with comment. The submittal of a Final RFI is not necessary. BASF need only submit an RFI Addendum that addresses the enclosed comments. Please submit the RFI Addendum within 60 days of the date of receipt of this approval letter.

As we discussed during our meeting on March 16, 1998, the Administrative Consent Order (Order), Docket V-W-94-R-011, calls for submittal of a Corrective Measures Study (CMS) Work plan within 60 days of the date of receipt of the RFI approval. However, based on BASF's concerns regarding the timing of the CMS Work plan submittal, additional ongoing and planned field and Risk Assessment efforts, and other long-term planning activities ("The Master Plan"), at the North Works site, the U.S. EPA is willing to consider a change in the CMS Work plan due date. Please submit a proposed schedule for submittal of the CMS Work plan within 60 days of receipt of this letter to the U.S. EPA for review and consideration.

The U.S. EPA understands that BASF has additional field efforts and investigations planned; focusing on AOC 4, AOC 5, and SWMU H, and the ground water extraction system. The U.S. EPA also understands that this additional work will lead to subsequent Risk Assessment additions, focusing on the Detroit River.

However, the U.S. EPA believes these additional efforts can easily be folded into the CMS.

Lastly, as a condition of RFI approval, the U.S. EPA is deferring the consideration of further investigation of releases from the North Works to the Trenton Channel or the Detroit River under this Order. The U.S. EPA does not intend to address contaminated sediments any further under this Order, but the U.S. EPA is reserving its rights under RCRA §3008(h), to address any releases and contaminated sediments in the future.

If BASF has any questions regarding the U.S. EPA's approval of the RFI, or this letter, please contact me at (312) 886-6199.

Sincerely,

Diane M. Sharrow, Project Manager  
U.S. EPA, Region 5  
Waste, Pesticides and Toxics Division  
Enforcement and Compliance Assurance Branch

Enclosure

cc: Thomas McGourty, BASF

bcc: Reginald Pallesen, ORC

**U.S. EPA COMMENTS ON BASF DRAFT PHASE 1 RFI  
(RFI APPROVAL WITH COMMENTS)**

**SECTION 1: DATA VALIDATION**

The data validation reports included in Appendix D of the RFI were reviewed. The purpose of the review was to determine the adequacy of the data validation performed by BASF's independent validation contractor, Environmental Standards, Inc., (ESI), in terms of completeness, accuracy and appropriateness of qualifications.

It appears that the validation was consistent with the requirements of the *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* and the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*. The validation effort appeared to be comprehensive and appropriately conservative, and included comments and observations that indicated that the data was thoroughly evaluated. Several minor transcription errors, and inconsistencies, as well as a limited number of technical errors were noted. However, in the vast majority of cases, it does not appear that correction of the technical errors will result in the rejection of any data currently deemed usable, since only "J" flags are affected in all cases except two (see Specific Comments 8 and 20).

**GENERAL COMMENTS:**

1. In several of the Data Validation Reports, sample results qualified as non-detected and flagged "U" were not further qualified as estimates and flagged "J" for QC deficiencies which required the qualification of non-detects. Examples include, but are not limited to, the following.
  - In Data Validation Report 5, *bis*(2-ethylhexyl)phthalate was qualified "U" due to blank contamination in samples SG005SWMUFSP07, SG010SWMUFSP06 and SG011SWMUFSP18. However, it appears that the results should have been qualified "UJ" to also account for low internal standard areas:
  - In Data Validation Report 7, antimony in sample SG002AOC6SP55 should be qualified "J" due to matrix spike recoveries, in addition to the "U" qualifier applied as a result of blank contamination.
  - In Data Validation Report 9, the antimony results for all samples should be qualified "J" due to matrix recoveries, in addition to the "U" qualifier applied due to blank contamination.

- In Data Validation Report 10, methylene chloride should be qualified "J" in samples SG008AOC23 and SG001SWMUG8 due to low surrogate recoveries, in addition to the "U" qualifier applied as a result of blank contamination.

BASF should revise the validation reports and all associated sections and tables of the RFI to reflect all changes in the qualifiers assigned in addressing this issue.

Alternatively, BASF should provide the rationale for not assigning those qualifiers associated with non-detects to results flagged "U" due to blank contamination.

2. It appears that the data reviewers were not consistent in the reporting of sample results on the Analytical Results tables for those results greater than the laboratory reporting limits and qualified as non-detects (flagged "U") due to blank contamination. According to the *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (Functional Guidelines)*, the reporting limit should be replaced by the detected concentration and qualified "U". For example, if a compound with a reported result of 10 ug/kg and a reporting limit of 5 ug/kg were qualified "U" due to blank contamination, the validated results should be 10 U, not 5 U. Revise the data validation as necessary to address this issue, ensuring that all affected validation reports, analytical summaries and Data Qualifier discussions are appropriately revised. If professional judgment was used to take an action other than that specified in the *Functional Guidelines*, provide a thorough rationale for this decision.
3. In those cases where samples were reanalyzed for organic parameters, the validation reports provide no indication of which set of results are of better quality and should, therefore, be used. In addition, in many cases the reanalyses were not clearly identified in the Analytical Results tables. Examples include, but are far from limited to:
  - In Data Validation Report 1, the results for sample SG001RFIMW24 and its reanalysis were validated and reported. It appears that the reanalysis may be the better of the two analytical runs, since all non-detects in the original acid fraction were rejected, but all results were qualified only as estimates (due to exceeded holding time) in the reanalysis.
  - In Data Validation Report 2, the results for sample SG013RFIMW07 and its reanalysis are reported. However, no reason is given for the reanalysis and, therefore, no determination as to which sample results to be used can be made.

- In Data Validation Report 5, the results for sample SG006SWMUFP11 and its reanalysis are reported. However, both samples exhibited the same QC criteria out of control, most likely indicating that the original analysis should be reported and used for decision-making purposes.

In each case where a sample was reanalyzed, revise all appropriate sections and/or appendices of the RFI Report to clearly indicate which analysis appears to be of better quality, based on the validation criteria, and to clearly indicate which set of results is reported and to be used for decision-making purposes.

4. Filtered and unfiltered inorganic results and field duplicate results were qualified based on poor agreement between the two sets of results for a given sampling location. Since validation based on the agreement between filtered and unfiltered results or field duplicate results are not addressed in the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, it appears that professional judgment was used to apply the qualifiers. Furthermore, it may be that either an unnecessarily conservative criterion was used, or errors exist in the application of qualifiers due to this issue. See for example the barium results for sample MW000RFIMW3 in Data Validation Report 25. Provide the rationale and criteria used to make all such judgments.

#### SPECIFIC COMMENTS:

1. Report 1 (SDG A6G1701300) - The Analytical Results tables for the inorganic analyses could not be located in Section 2, Analytical Results. Based on the text, it appears that validation was appropriately performed, however, no verification of the assignment of qualifiers could be made. Provide the missing tables.
2. Report 2 (SDG A6G250145) - According to Item 11 of the Organic Data Qualifiers in Section 1, the volatile organic compounds (VOCs) quantitated using the internal standard chlorobenzene-d5 were qualified in sample SG004RFIMW07. However, the qualifiers were not included on the Analytical Results table on page 1 and 2 of Section 2. Revise the Data Validation Report to address this discrepancy.
3. Report 4 (SDG A6310114) - Item 1 of the Organic Data Qualifiers in Section 1 incorrectly indicates that pentachlorobenzene has been rejected. As accurately reflected in the Analytical Results table in Section 2, the

compound for which the result was rejected is actually pentachloronitrobenzene. Revise the Data Validation Report to address this discrepancy.

4. Report 5 (SDG A6H020118) - For sample SG010SWMUFSP06, the compound chloroethane, rather than chlorodibromomethane, should have the "UJ" qualifier on page 10 of the Volatile Analytical Results table in Section 2. Revise the Data Validation Report to address this transcription error.
5. Report 5 (SDG A6H020118) - For sample SG006SWMUFSP11, the compound pentachloronitrobenzene, rather than pentachlorophenol, should have the "UR" qualifier on page 19 of the Semivolatile Analytical Results table in Section 2. Revise the Data Validation Report to address this transcription error.
6. Report 6 (SDG A6H030119) - The pH results are not included in the Analytical Results tables in Section 2. Revise the tables to include the pH results.
7. Report 7 (SDG A6H150150) - Sample RS000AOC6 was incorrectly identified as AS000AOC6 in Item 2 of the Organic Data Qualifiers section. Correct this typographical error.
8. Report 8 (SDG A6H160167) - The Data Validation Report does not address the low recoveries for surrogate TCX and the zero percent recoveries for DCB reported on Form 2 for sample SD004SWMUE4 in the pesticide/PCB results. It appears that all non-detected results for this sample should be rejected. Either provide a thorough rationale for not rejecting the results, or revise as necessary all affected sections of the Data Validation Report and the text of the RFI Report to address the rejection of these results.
9. Report 8 (SDG A6H160167) - Item 2 of the Organic Data Qualifiers section identifies an incorrect sample number. The actual sample qualified appears to be SD003SWMUE1, rather than SD004SWMUE4, as reflected in the Analytical Results tables. Revise the Data Validation Report to address this discrepancy.
10. Report 8 (SDG A6H160167) - According to the Volatiles Analytical Results tables, 2-butanone (MEK) was qualified as "UJ" in samples SD003SWMUE2 and SD004SWMUE4. However, the Organic Data Qualifiers section does not address this compound. Revise the Data Validation Report to address this inconsistency.
11. Report 9 (SDG A6H160149) - 1,2-Dichloroethane was rejected and flagged "R" in samples SG002AOC6SP63 and SG002AOC6SP64 according to the Volatile Analytical Results tables. However, the Organic Data Qualifiers section provides no

reason for these qualifiers. Revise the Data Validation Report to address this issue.

12. Report 10 (SDG A6H220161) - Several VOCs were apparently incorrectly qualified for low internal standard areas based on a review of relative retention times found on page 378 of the associated data package. Tetrachloroethene, toluene and 4-methyl-2-pentanone should also be qualified "UJ" for those samples qualified due to low chlorobenzene-d5 areas. In addition, EDB and chlorodibromomethane are not associated with chlorobenzene-d5, but with internal standard 1,4-difluorobenzene and should not, therefore, be qualified in those samples with chlorobenzene-d5 as the only internal standard with areas outside the QC limits. Revise the Data Validation Report to address this issue.
13. Report 10 (SDG A6H220161) - The compound phenacetin was qualified "UJ" in the Semivolatile Analytical Results tables for sample SS001SWMUG4. However, the Organic Data Qualifiers section does not address this qualifier. Revise the Data Validation Report to correct this discrepancy.
14. Report 10 (SDG A6H220161) - It is indicated under the Noncorrectable Deficiency, Item 4, listed on page 4 of Section 1, that the impact of the deficiency is discussed in the Organic Qualifier Section. However, said discussion could not be found, nor were qualifiers applied to the associated compound, methoxychlor. Revise the Data Validation Report to provide this information and any associated qualifiers.
15. Report 11 (SDG A6H240102) - It is indicated in the Inorganic Data Qualifiers section that the zinc result for sample SG001AOC26 should be qualified "J". Revise the Analytical Results table to include this qualifier.
16. Report 12 (SDG A6H240106) - It appears that the zinc result for sample RS00AOC7, an aqueous sample, was incorrectly qualified based on the results of the soil ICP serial dilution. Revise the Data Validation Report as necessary to address this issue.
17. Report 13 (SDG A6H270124) - All results for sample RS000AOC2 were qualified "UJ/J" because the sample was analyzed one day beyond the seven-day holding time. According to the *Functional Guidelines*, only aromatic compounds should be qualified in unpreserved aqueous samples when the seven-day holding time is exceeded. All other compounds have a 14-day holding time. (The Data Validation Report does not clearly indicate that the sample was unpreserved; it is only so inferred based on the application of the qualifiers). Revise the Data Validation Report as necessary to address

this apparent unnecessarily conservative application of qualifiers.

18. Report 15 (SDG A6I100127) - It is indicated in the Organic Data Qualifiers section that the 1,3,5-trinitrobenzene result for sample SW000AOC7 should be qualified "UR". Revise the Semivolatile Analytical Results table to include this qualifier.
19. Report 24 (SDG A6I250139) - According to Item 4 of the Organic Data Qualifiers section, the 4-nitrophenol results should be qualified in two of the four reported samples. However, the results for this compound are qualified in all four samples on the Semivolatile Analytical Results tables. Revise either the text or table as necessary to address this discrepancy.
20. Report 30 (SDG A6J010134) - The second item of the Noncorrectable Deficiencies section indicates that the holding times were exceeded by 20 days for samples MW000RFIMW-29, MW000RFIMW30 and TB-10/1/96. For these samples, the holding times have been grossly exceeded and all VOC non-detect results should be rejected and flagged "R". Either revise all associated sections and tables of the Data Validation Report and main RFI Report to address these rejections, or provide a thorough rationale for deviating from the *Functional Guidelines*.
21. Report 30 (SDG A6J010134) - The second item in the Inorganic and Wet Chemistry Data Qualifiers section indicates that the cyanide samples were of a soil matrix. However, the Analyte Results Summary and supporting documents indicate a water matrix. Revise the Data Validation Report to address this discrepancy.
22. Report 32 (SDG A6K200114) - All VOC results, except those previously flagged "UR", were qualified as estimated due to exceeded holding times. However, it is indicated in Item 5 of the Organic Data Qualifiers section that only the aromatic compounds were qualified. Revise the Data Validation Report to resolve this discrepancy.
23. Report 33 (SDG A6L190128) - The analytical results table for the VOC analysis of sample MW000RFIMW9 was not included in Section 2, Analytical Results. While the reported validation appears to be accurate, the application of qualifiers could not be confirmed. Revise the Data Validation Report to include this table.
24. Report 33 (SDG A6L190128) - Several results were rejected and flagged "R" due to very low Laboratory Control Sample (LCS) recoveries. However, validation due to LCS results are typically based on project-specific criteria. No such



criteria are presented. Revise the Data Validation Report to discuss the criteria used to qualify results based on LCS recoveries.

25. Report 34 (SDG A7C190143) - Several semivolatile results were rejected and flagged "R" due to very low Laboratory Control Sample (LCS) recoveries. However, validation due to LCS results are typically based on project-specific criteria. No such criteria are presented. Revise the Data Validation Report to discuss the criteria used to qualify results based on LCS recoveries.
26. Report 35 (SDG A7F030144) - It appears that Item 7 in the Inorganic Data Qualifier section incorrectly identified the qualifier due to RPD criteria as "U\*". The qualifier should be "UJ" unless previously qualified due to blank contamination. Revise the Data Validation Report to address this apparent discrepancy.

## SECTION 2: SPLIT SAMPLING

### NOTE:

Intertek Testing Services (ITS), of Richardson, Texas, a subcontractor laboratory to TechLaw, Inc., (TechLaw), a U.S. EPA Contractor, recently notified the U.S. EPA of irregularities in its data reporting for volatile organic compound (VOC) analyses using methods under SW-846 for testing soils and waters. ITS has reported that these irregularities involved the inappropriate manual integrations of chromatographic peak areas for the purpose of meeting method/protocol criteria.

All samples obtained at the North Works by TechLaw were split samples and, therefore, meant to confirm BASF's laboratory's performance. As is discussed in the comparison of split sample results, the results obtained by TechLaw and BASF are generally comparable and provide no indication of poor performance by BASF's laboratories.

Analytical results from the RFI and U.S. EPA's split sampling activity were compared for the concentrations of detected constituents. In summary, analytical results obtained by BASF's laboratory (Quanterra) and U.S. EPA's Contractor's (TechLaw) laboratory (ITS) appear to be generally comparable.

The split sample results are shown in Table 1 (Comparison of Groundwater Sample Results) and Table 2 (Comparison of Soil Sample Results). As can be seen upon review of these tables, the laboratories generally identified the same compounds as present in the samples. When the same constituents were detected, the relative percent difference (RPD) results ranged from a low of 1% to a high of 153%. In most cases, the RPD was below 50%, a

standard criterion for comparing a sample and its duplicate. While the results presented in Tables 1 and 2 do show moderate to high variability between the laboratories for certain constituents, the range of results appears to be within acceptable limits for a split sampling event, considering that the samples were analyzed by different laboratories using different instruments and Standard Operating Procedures (SOPs). In addition, the types of compounds detected by each laboratory are similar (primarily polynuclear aromatic hydrocarbons) and all variations in split sample results are by less than an order of magnitude.

Several organic compounds were reported by one laboratory but not the other. In many cases the detected concentration was close to or below the detection limit for the other laboratory. See for example the phenol results reported for sample MW000RFIMW2. In addition, the vast majority of the remaining compounds detected by one laboratory but not the other are common laboratory contaminants, such as methylene chloride and phthalate compounds. All organic compounds for which positive results were reported by either laboratory are included in Tables 1 and 2.

Not all inorganic constituents are included in Table 2. Metals concentrations vary considerably depending upon sample location, media and the aliquot chosen for analysis. Therefore, only those inorganic constituents detected at concentrations above the BASF Project Specific Action Levels (PSALs), as noted in the RFI, are included for comparison. Additional inorganic constituents were detected in the groundwater and soil samples but have not been included here.

Two field duplicate samples were obtained by TechLaw during the split sampling activities. The water samples RFIMW15 and RFIMW115 are included in Table 1 and soil samples SS001SWMUG-5 and SS001SWMUG-105 in Table 2. As with the split samples, the agreement between the field duplicates was generally acceptable, with limited instances of higher degrees of variation in the soil samples.

**TABLE 1 - COMPARISON OF GROUNDWATER SAMPLE RESULTS**

ANALYTES	BASF RESULT (ug/L)	TECHLAW RESULT (ug/L)	RPD
<b>MW000RFIMW2</b>			
Acetone	27	10.0 U	NC
Carbon Disulfide	0.59	1.0 U	NC
bis (2-Chloroethyl) ether	45	47.4	5.4%
1,4 Dioxane	4	500 U	NC
4-Methylphenol	95	88.9	6.6%
3-Methylphenol	95	Not reported	NC
Phenol	5.3	10.0 U	NC
<b>RFIMW15 <sup>1</sup></b>			
bis (2-Ethylhexyl) phthalate	5 U	5.16 J / 10 U	NC / NC
bis (2-Ethylhexyl) phthalate (Reanalysis)	5 U	10 U / 10 U	NC / NC
<b>MW000RFIMW16</b>			
Acetone	540	328	49%
Benzoic Acid	Not reported	29.0 J	NC
Carbon Disulfide	3.4	2.9	16%
1,4, Dioxane	39	500 U	NC
Methyl Ethyl Ketone	43	10.0 U	NC
4- Methylphenol	200 U	11.6	NC
Phenol	350	228	42%
Toluene	2	1.0 U	NC

U = Not Detected at shown Quantitation Limit

NC = Not Calculated

<sup>1</sup> The semivolatile analysis was rerun to improve identification of numerous Tentatively Identified Compounds (TICs) detected during the original analysis. The TICs are not addressed here, but are further identified in Form 1 SV-TIC data sheet number 223 in the original data package. Results and RPD are for a primary environmental sample (RFIMW15) and a duplicate environmental sample (RFIMW115) respectively.

ANALYTES	BASF RESULT (ug/kg)	TECHLAW RESULT (ug/kg) <sup>1</sup>	RPD <sup>1</sup>
<b>SS001SWMUG-5</b>			
Acenaphthylene	150 J	330 U / 120 J	NC / 22%
Anthracene	90 J	330 U / 81.4 J	NC / 10%
Benzo(a)anthracene	440	492 / 647	11% / 38%
Benzo(b)fluoranthene	790	947 / 997	18% / 23%
Benzo(k)fluoranthene	220 J	355 / 371	47% / 51%
Benzo(g,h,i)perylene	220 J	404 / 463	59% / 71%
Benzo(a)pyrene	420	558 / 586	28% / 33%
Chrysene	440	628 / 734	35% / 50%
Dibenzofuran	210 J	272 J / 330 U	26% / NC
Fuoranthene	510	648 / 734	24% / 36%
Indeno(1,2,3-cd)pyrene	200 J	396 / 409	66% / 69%
2-Methylnaphthalene	600	995 / 667	50% / 11%
Naphthalene	360 J	656 / 425	58% / 17%
Phenanthrene	540	770 / 622	35% / 14%
Pyrene	540	691 / 747	25% / 32%
Arochlor 1260	370 J	398 / 430	7% / 15%
Pentachlorophenol	39	1,650 U / 1,650 U	NC / NC
Arsenic	65.6 (12)	56.7 / 80.9	15% / 21%
Lead	121 (63.3)	86.1 / 85.1	34% / 35%
Methylene Chloride	6 U	12.0 / 8.6	NC / NC
Di-n-butylphthalate	370 U	330 U / 43.4 J	NC / NC
bis(2-ethylhexyl)phthalate	370 U	330 U / 108 J	NC / NC
Dibenz(a,h)anthracene	370 U	330 U / 149 J	NC / NC
beta-BHC	9.4 U	5.10 J / 1.50 U	NC / NC
Heptachlor	9.4 U	5.60 J / 5.00 J	NC / NC
2,4-D	110 U	30.6 J / 23.3 J	NC / NC

ANALYTES	BASF RESULT (ug/kg)	TECHLAW RESULT (ug/kg) <sup>1</sup>	RPD <sup>1</sup>
<b>SG002AOC2-5</b>			
Methylene Chloride	10 U	11.0	NC / NC
Chromium	101 (23.9)	13.4	153%
Mercury	17.1 (0.8)	17.0	1%
Total Cyanide	21 (0.1)	7.8	92%

U = Not Detected at shown Quantitation Limit

NC = Not Calculated

<sup>1</sup> Results and RPD are for a primary environmental sample (SS001SWMUG-5) and a duplicate environmental sample (SS001SWMUG-105) respectively

## SECTION 3: HYDROGEOLOGICAL ASSESSMENT

### GENERAL COMMENTS:

1. Groundwater samples collected from monitoring wells RFIMW-5, RFIMW-6, RFIMW-7, and RFIMW-12 have historically had pH levels greater than 12. BASF should discuss the high pH and the significance in the RFI Addendum.
2. The quantification limits for the various semi-volatile and metal analysis run on soil samples from the well borings for the seven background monitoring wells are significantly different than the quantification limits for the analysis run on the background samples for fill and sand (Tables 7-1 and 7-2). BASF should discuss with Quanterra and discuss in the discrepancy in the RFI Addendum.
3. On many of the potentiometric maps located in the RFI, information overlaps other information, making both illegible. BASF should revise the applicable figures and maps to eliminate overlap.

### SPECIFIC COMMENTS:

1. Section 3.1.1, Site Geology, Pages 3-1 and 3-2. BASF refers to prior literature evaluations and subsurface investigations, but does not specifically identify these sources. Please include this information in the RFI Addendum.
2. Section 7.1.1.3, Geological Cross-sections, Page 7-4. BASF should elaborate in the RFI Addendum on how the cross-sections corroborate the unit descriptions.
3. Section 7.1.2.2, Groundwater Elevation Data, Page 7-7. BASF should clarify in the RFI Addendum how other evaluations and documentation support the discussion on groundwater flow at the Facility.
4. Section 7.1.1.3, Step Drawdown Test Results, Pages 7-10 and 7-11. BASF should add references to sections 7 and 9 of the RFI to the RFI Addendum that supports Facility's contention that the Peat and Clay unit act as an aquitard and a confining layer, and that shallow groundwater is divided into two distinct units.

5. Page 7-11. BASF does not indicate the direction of flow on any of the figures as is typically done with flow lines and/or arrows. The RFI addendum should include revisions of these figures.

6. Section 7.1.2.4, Estimation of Capture Zones, Pages 7-11 and 7-12. BASF should clarify in the RFI Addendum text the differences between Figures C-10 and 7-15.

7. Section 7.1.4, Evaluation of Groundwater Extraction System Efficiency, Page 7-13. BASF should map the clay ridge divide in the RFI Addendum ; without the presence of flow lines it is difficult to determine where BASF believes the divide or divides to exist.

8. Section 7.4.4, Summary of Groundwater Analytical Results. Additional discussion of the effectiveness of the Groundwater Extraction System to capture contaminated groundwater, as well as any shortfalls, should be summarized in the RFI Addendum, with more extensive discussion to be provided in the Corrective Measures Study.

From: LARRY KYTE  
To: KUJAWA-JEROME, PALLESEN-REGINALD, TANAKA-DEIRDRE, ...  
Date: 5/4/98 11:18am  
Subject: ITS Discussions

BASF

As most of you are aware of by now, I am the Regional liaison regarding ITS matter. You all happen to be on RCRA case teams that have been blessed with having samples reportedly analyzed at the Richardson laboratory of ITS that has been the subject of an investigation resulting from the company's self-disclosure.

As the investigation continues there is likely to be discovery of other instances of use of the laboratory related to EPA authorities. These also will need to be investigated to determine the reliability of the data used. So you may only be the first.

Each team has by now made an assessment of the impacts of ITS samples results at its own site. The data from the Richardson lab for your samples may not be deemed of significant importance to your investigations. However, in each instance you eventually will need to determine whether, when and how you will inform the Respondents and the public how these samples impact your reliance on those samples. It is likely that written disclosure to the Respondent or defendant, which will be in a public file (administrative or court record) may constitute sufficient notice to the public as all of the your current cases are under investigation and no final decisions have been made.

Prior to any written correspondence with a party outside of the federal government, I am requesting that you provide me notice and an opportunity to confer and comment. Because the matter is under investigation, and its final scope is uncertain, we should be ~~hesitant to~~ comment beyond the fact that ITS has submitted a self-disclosure notice of certain improper activities in its Richardson, TX laboratory and that as a result of those disclosures we are conducting an investigation. We should refrain from making any statements that have reliance on any promises by the laboratory to make good and correct the problem or reanalyze the data at this time.

I am providing each of the ORC attorneys assigned to these cases with copies of the Self-disclosure statement, a January 30 letter from ITS to one of its customers (TechLaw), and a copy of a memo from Sylvia Lowrance dated April 13, 1998 which sets out certain protocols regarding contacts and protocols for disclosure.

CC: its



**From:** DIANE SHARROW  
**To:** PHILLIPS-GERALD, LITTLE-PAUL  
**Subject:** 4/6/98 8:51am  
CA & RCRIS

BASF

Gerry and others,

With the focus being placed on using RCRIS for planning and reporting for CA, I want to raise to your attention (again), a long-running problem. BASF, North Works MID 064 197 742 is one of my sites with a RCRA 3008(h) Order.

If you check RCRIS you will see that the site is called BASF Pte. Hennepin. This is incorrect. Pte. Hennepin is on Grosse Ile. BASF North Works is on the mainland at 1609 Biddle Ave.

Pte. Hennepin does not have an address - however, it does have an ID number that was assigned by the State when BASF did a voluntary action. The ID Number for Pte. Hennepin is MIO 000 938 704.

I am not sure whether it is a problem with RCRIS that the State has to use the Biddle Avenue ID no to report its activities because Pte, Hennepin never had interim status, or what. The end result is that the information in RCRIS under MID 064 197 742 is contradictory and misleading due to this double reporting by Michigan's 201 program.

I talked to Larry Aubuchon at DEQ-Livonia about doing something to correct this situation, but never heard back.

I do not want to do any "clean-up" on the information there until this problem is fixed.

What do I need to do to get the names and information corrected in RCRIS?

Paul Little suggested contacting Sharon Goble at DEQ. What do you "guys" feel needs to be done to correct this problem once and for all.  
Diane Sharrow, USEPA, Region 5  
Waste, Pesticides & Toxics Division  
Enforcement & Compliance Assurance Branch  
77 W. Jackson Blvd., DE-9J  
Chicago, IL 60604  
Facsimile 312.353.4342  
Phone/Voice Mail 312.886.6199  
E-MAIL: Sharrow.Diane@epamail.epa.gov

**CC:** BAKK-DANIEL, R5CHG.IN("GobleS@state.mi.us")

**From:** DANIEL BAKK  
**To:** LITTLE-PAUL, PHILLIPS-GERALD, SHARROW-DIANE  
**Date:** 4/6/98 9:00am  
**Subject:** CA & RCRIS -Reply

Diane, these are important corrections that need to be made in RCRIS. LaNita Marrable is our RCRIS State coordinator with MI. She is the appropriate person to follow-up on these issues.

>>> DIANE SHARROW 04/06/98 08:51am >>>

Gerry and others,

With the focus being placed on using RCRIS for planning and reporting for CA, I want to raise to your attention (again), a long-running problem. BASF, North Works MID 064 197 742 is one of my sites with a RCRA 3008(h) Order.

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Diane Sharrow, USEPA, Region 5  
Waste, Pesticides & Toxics Division  
Enforcement & Compliance Assurance Branch  
77 W. Jackson Blvd., DE-9J  
Chicago, IL 60604  
Fascimile 312.353.4342  
Phone/Voice Mail 312.886.6199  
E-MAIL: Sharrow.Diane@epamail.epa.gov

CC:

R5CHG.IN."GobleS@state.mi.us", MARRABLE-LANITA, RA...

**From:** DIANE SHARROW  
**To:** MARRABLE-LANITA  
**Date:** 4/6/98 9:15am  
**Subject:** CA & RCRIS -Reply -Forwarded

LaNita,

Anything you can do to help me with the attached would be appreciated.

Thanks

Diane Sharrow, USEPA, Region 5  
Waste, Pesticides & Toxics Division  
Enforcement & Compliance Assurance Branch  
77 W. Jackson Blvd., DE-9J  
Chicago, IL 60604  
Fascimile 312.353.4342  
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E-MAIL: Sharrow.Diane@epamail.epa.gov

**CC:** LITTLE-PAUL, PHILLIPS-GERALD, SHARROW-DIANE

CERTIFIED MAIL - RETURN RECEIPT REQUESTED

P 633 102 350

March 2, 1998

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (DRE-9J)  
77 West Jackson Street  
Chicago, Illinois 60604

Subject: Responses to TECHLAW Data Inquiries for Draft Phase I RCRA Facility Investigation Report  
Docket No.: V-W-011-94  
BASF Corporation, Wyandotte, Michigan

Dear Ms. Sharrow:

BASF Corporation is submitting this letter in response to data inquiries involving the above-referenced draft report dated December 4, 1997. The inquiries posed by Mr. John Koehnen of TECHLAW and our associated responses are summarized below.

- Q1: TECHLAW inquired about the availability of supplemental boring logs for piezometers that were referenced in the report cross-sections, but not included within Appendix B (Soil Boring and Monitoring Well Logs).
- R1: BASF is enclosing copies of well logs. The associated piezometer installation activities (PE1NA, PE4NA, PE10NB, PE13NB, and PE14NC) were completed as part of the ancillary pump test activities, not part of the originally scoped RFI tasks, and were prepared by Jack Lanigan of BASF. DNR 4 and DNR 6 were installed in 1981 by the Michigan Department of Natural Resources and were originally labeled as BW4 and BW6 respectively. Piezometers P4N, P12N, P27N, P34N, P37N, and P39N were installed by S. S. Papadopoulos & Associates in the early 1980s. As a result, the log format appears different from those prepared by QST Environmental.

Data from historic DNR- and P- series piezometers/wells were utilized in the cross-section figures for lithological completeness purposes only. These monitoring locations were not installed during the scoped RFI activities, hence were not included within Appendix B. For the revised report, a reference notation will be incorporated into each cross-section figure to denote these pre-RFI boring locations.

- Q2: TECHLAW inquired whether a groundwater "profile" had been prepared.
- R2: Groundwater profiles were not part of the QAPP. Therefore none were prepared. Groundwater contour plots supplied within the report depict groundwater flow patterns at the Facility.
- Q3: TECHLAW inquired about the availability of additional groundwater elevation values for various monitoring wells.
- R3: BASF is enclosing supplemental groundwater elevation data for 10 wells in September 1996 that were measured one time. The four quarterly monitoring events in the form of Tables E-7 through E-10 have also been re-formatted to facilitate easier data comparison between the quarterly events.
- Q4: TECHLAW indicated an interest in reviewing additional pump test-related drawdown data associated with the adjacent piezometers PE2NA, PE13NB, and PE14NC.
- R4: BASF is enclosing supplemental drawdown data for these piezometers. The tabular data include transducer readings for the pumped well and the associated piezometer, but do not include actual groundwater elevation values. The tabular data were used in determining flowrate values presented in the step-drawdown test analysis summaries for each of the three pumped wells (see Tables C-1, C-2, and C-3 of the Draft Report).

Site-specific complexities (e.g. extremely heterogeneous lithology, potential existence of two separate water-bearing units, effects of existing groundwater extraction system, etc.) influenced the pump testing evaluation methods and associated goals. As a result, pump test conclusions were focused on assessing the efficiency/performance of the existing groundwater extraction system.

A copy of the enclosures is being sent separately to TECHLAW's Mr. Steve Phillips in Texas.

Should you need additional information, please contact us at your convenience.

Sincerely,



K. Edward Nuernberg  
General Manager

Enclosures

z:\zword\admin\techlaw.doc

cc: B. Roberts - BASF  
D. Marian - QST  
R. Blayer - MDEQ Lansing  
L. Aubuchon - MDEQ Livonia  
J. Russell - MDEQ Livonia  
A. Danford - Quanterra (letter only)  
B. Wallace - Bacon Memorial Public Library

TABLE 2-2

COMPOUNDS IDENTIFIED IN SOIL OR GROUNDWATER  
DURING 1981 INVESTIGATION

2-Ethylhexanol  
Ethylpyridine  
- Fluorene  $\Delta$   
- Fluoranthene  $\Delta$   
- Hexachlorobutadiene  $\Delta$  \*  
- Indene  
- Indane  
Isopropylbenzene  
Methylaniline  
Methylene Chloride  
- Methylnaphthalene *is this 2-methylnaphthalene*  
Methylphenylacetylene  
Methylstyrene  
2-Methylthiophene  
- Naphthalene  $\Delta$   
Nonylphenol  
- Pyrene  
Phenyl Acetic Acid  
- Lead  $\Delta$   
Pyridine  
Phenyl Ethyl Acetic Acid  
Phenol  
- Phenanthrene  $\Delta$   
- Styrene  $\Delta$   
- Toluene  $\Delta$   
Trichlorobenzene  
Tetrachlorobutadiene  
Toluenediamine  
Trimethylbenzene  
Tetramethylsuccimonitrite  
Thiophene  
- Xylene  $\Delta$   
Xanthene  
- Zinc  $\Delta$

$\Delta$  App IX Const.



← ←

↪



Diane,

Attached is a DRAFT agenda, please comment. After you comment on it, I will route it around for comments from site folks and QST.

I have a 10:00 AM starting time because I assume you would fly in that morning. Feel free to adjust the starting time to suit your schedule.

9:00 am

I sent out an E-Mail note concerning a date and location change.

We can have lunch brought in for everyone. It is usually easier that way. ~~Most of the time they have fried chicken, potatoes, and green beans and cookies for desert. If you prefer, we can break for lunch with everyone going their separate ways. I do not want to raise any potential conflict of interest issues. Please advise which way you prefer to do lunch.~~ <sup>OK</sup>

You are welcome to tour the site again. — Yes

My FAX number is (313) 246-6774.

Bruce Roberts

**AGENDA****BASF - EPA MEETING TO DISCUSS DRAFT RFI INVESTIGATION REPORT****\* MONDAY MARCH 16, 1998****9:00 ~~10:00~~ - 4:30 ~~4:00~~****CONFERENCE ROOM \_\_, SITE ADMINISTRATION BUILDING****I. INTRODUCTIONS** (EPA Team will be available by phone)**II. DISCUSSION OF DRAFT REPORT****A. OVERVIEW - BASF****B. OVERALL IMPRESSIONS INCLUDING CONCERNS -- EPA****C. OPEN ISSUES -- ALL****1. SITE HYDROGEOLOGY****2. DATA QUALITY OBJECTIVES****DATA VALIDATION****3. RISK ASSESSMENT****Letter of 2/12/98****4. SEDIMENTS****5. OTHER CONCERNS****III. NEXT STEPS IN THE CORRECTIVE ACTION PROCESS****A. CONSENT ORDER & RCRA CORRECTIVE ACTION  
PROCESS SEQUENCE OF EVENTS -- EPA****B. SEQUENCE OF EVENTS AS IT APPLIES TO BASF -- ALL****IV. OTHER ISSUES?****V. SITE TOUR/INSPECTION****\* I was approved for travel 2/26.****Bruce - Could you have entire RFI at meeting?  
I will only bring selected pieces? Thanks****ITS  
Lab  
Probs.**

**From:** Arthur Ostaszewski <OSTASZEA@state.mi.us>  
R5WST.R5RCRA(SHARROW-DIANE)  
**Date:** 2/9/98 2:26pm  
**Subject:** Draft BASF- Northworks Phase I RFI Report -Reply -Reply -Reply

Proving contamination is "anybodys in particular" has been one of the major difficulties in remediating the sediments in the Trenton Channel. Thats why we embarked on a system-wide study of sediment contamination in the Trenton Channel, to pick out spikes where the levels elevate as one goes downstream. The other impediment to remediation being what to do with it, (disposal options).

In looking at the other sediment areas we sampled upstreams and around the Northworks site, we found very little contamination upstream at Mud Island and Grassy Island, but found higher contamination at the Stenson Club, (near the head of the Trenton Channel-proper and National Steel).

Basically, it appears from our study that sediments are generally less contaminated immediately upstream of BASF Northworks, and show an increase at and below the facility.

Not to discount National Steel probable play in this, If BASF claims the sediment contamination is not their's, where is thier proof? Since there is some spike in contamination in front and downstream of their facility, I think the burden would be on them to make a good faith effort to clean it up to below Low Effect Levels. If they think they can get other prps involved to share the costs, thats better for them.

I remember our core from in front of BASF very well, it took several tries but we found the patch of soft mud. The area around BASF is a high energy area and not prone to deposition. Not being familiar with the RFI, did BASF adaquately map out soft vs hard sediment areas around the Northworks facility? I imagine any sediment remediation in the zone in front of BASF Northworks would be under 20,000 Cubic Yards. The area downstream at the Wyandotte Yacht Club maybe 50,000 cubic yards, (these are rough estimates).

There is no "smoking gun" tracer parameter that we can tie to Northworks, or any other potential PRP. Its just a spike in contaminant levels as compared to upstream and surrounding areas.

Thanks for keeping me in the loop.

Art

Art Ostaszewski  
MDEQ/SWQD-GLEAS

**From:** Arthur Ostaszewski <OSTASZEA@state.mi.us>  
R5WST.R5RCRA(SHARROW-DIANE)  
**Date:** 2/9/98 10:18am  
**Subject:** Draft BASF- Northworks Phase I RFI Report -Reply -Reply

Concerning Sediments near BASF Wyandotte Northworks:  
Based on Trenton Channel Project Sampling, we took one core offshore at the south end of the property, and one core in the closest depositional zone downstream of the facility.

A summary of the data reveals the following:

BASF Northworks-lower site (all values-ppm d.w.)

interval	PCBs	PAHs	Oil and Grease	SEL metals
00-30 cm	1.93	32.1	11000	Pb Ni Zn
30-91	1.30	37.2	10000	Cd Pb Hg Ni Zn
91-152	0.42	88.1	8000	Cd Pb Hg Zn
152-213	<.3	87.7	4000	Zn
213-218	<.3	6.4	<50	

SEL = Severe Effect Level

Wyandotte Yacht Club (mg/kg d.w.)

interval	PCBs	PAHs	Oil and Grease	SEL metals
00-30cm	3.29	33.4	9000	Pb Zn
30-66	1.77	32.1	9000	Pb Hg Zn

Our site above BASF Northworks showed the following:

Mouth of Ecorse Creek (mg/kg d.w.)

interval	PCBs	PAHs	Oil and Grease	SEL metals
00-30	1.11	< 0.33	<50	
Ponar	3.49	25.9	8000	Zn

I have not seen the RFI, Our data shows an increase in sediment contaminant levels for PAHs, Oil and Grease, Cd, Pb, Hg, and Ni comparing upstream sediments to those across and downstream of the Northworks facility.

Matt Williams has a copy of our Trenton Channel Sediment Report, published July 1997. These levels are high enough to impact biota, though as Matt points out, do not trigger any specific enforcement action. Does BASF propose any remedial action for the sediments?

In the Trenton Channel Sediment Report we identify much bigger fish to fry in Trenton Channel than this site (contaminant levels, volume of sediment). Unless the RFI or your sampling has showed much higher levels than we had, I see SWQD continuing our investigation of the more contaminated sediments sites at this point in time. Once those sites are remediated, we would then start on this site. Ive always had good relations with BASF. If they are interested in being pro-active and addressing the sediment contamination off the Northworks Plant now, we are willing listen and help.

If you would like a copy of our Trenton Channel Sediments Report (84 stations), you can wait until it makes the best sellers list in paperback, or I could send you an autographed copy right now. Let me know.

Thanks,

*Copsey*  
*Engel*  
*GTB*  
*Availability*  
*September*

Art Ostaszewski  
SWQD-GLEAS

**From:** MATTHEW WILLIAMS  
**To:** R5WST.R5RCRA.SHARROW-DIANE, R5AIR.R5ARD.OLENDER-MA...  
**Date:** 2/7/98 2:13pm  
**Subject:** Draft BASF- Northworks Phase I RFI Report

Marc and Diane:

As requested, I have reviewed BASF's (Northworks) draft Phase I RFI report with respect to contaminated sediment in the Trenton Channel adjacent to and near the facility's property.

They (and their contractor QST) did a decent job summarizing the current contaminated sediment data for the Trenton Channel. Although this isn't too hard considering MDEQ, USEPA GLNPO and Region 5 have done considerable sampling there over the past six years. Still, it was a pretty good summary.

RFI Sections 6 and 7 discuss sediment contamination near the facility. Adjacent to the facility, the water is deep and fast. There is really not much sediment to sample. So they are correct in that assessment. I have tried collecting sediment from that area with little luck - mostly rock and gravel with some sand. Both upstream and downstream of the facility, there are pockets of sediment that have been sampled recently. Results show PCBs, PAHs and metals at levels high enough to impact biota, but too low to trigger any heavy hammers (i.e., Superfund Removal or RCRA Interim Measures). Still, the stew of contamination is not good to just leave alone. I know MDEQ and GLNPO have prioritized these "hot pockets" in the RAP and in other reports in pursuit of funds to start cleaning up the feasible areas. Presently, I do not have information to suggest that BASF northworks is solely responsible for this contamination.

Unless new data comes forward, I believe that BASF should not use resources to conduct additional sediment sampling in the Trenton Channel. Instead, these resources should be added to better characterize soil and/or groundwater contamination on the facility - to protect from future contamination by the facility of the channel and its sediment.

Art Ostaszewski MDEQ (517.335.4491) is knows the channel and its sediment like the back of his hand. If you haven't already, I would discuss this matter with him as well.

Thanks

Matt (3-4934)

**CC:** art,arto

January 22, 1998

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (DRE-9J)  
77 West Jackson Street  
Chicago, Illinois 60604

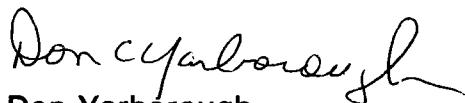
Dear Ms. Sharrow:

Subject: Letter of Errata for Draft Phase I RCRA Facility Investigation Report  
Docket No.: V-W-011-94  
BASF Corporation, Wyandotte, Michigan

BASF Corporation is submitting this letter in association with the above-referenced document dated December 4, 1997. Within Appendix B of the document (Soil Boring and Monitoring Well Logs), the monitoring well logs should reflect that the well casing materials consisted of stainless (and not galvanized) steel. Conversely, the term "stainless" has been deleted from the split spoon sampler description for each boring log. These revisions will be incorporated with additionally required agency modifications and submitted as part of the Final RFI Report.

Should you need additional information, please contact us at your convenience.

Sincerely,



Don Yarborough  
Wyandotte Site Manager

z:\zword\admin\vw01194.doc

cc: B. Roberts - BASF  
D. Marian - QST  
R. Blayer- MDEQ Lansing  
L. Aubuchon - MDEQ Livonia  
J. Russell - MDEQ Livonia  
A. Danford - Quanterra  
B. Wallace - Bacon Memorial Public Library

**MEMORANDUM**

**DATE:** 9 December 1997

**SUBJECT:** BASF, Inc., MID 064 197 742

**FROM:** Diane Sharrow, Project Manager

**TO:** Gerald Phillips, Process Manager

I am proposing to change BASF's schedule dates in RCRIS / CARS for the following event codes 200, 400, 500 and 550. The Facility submission of the RFI has been delayed due to a number of events, including problems with existing of wells, replacement of wells, and the Region's desire for full Appendix IX sampling based on the history of the Facility and its proximity to the Detroit River. Specifically, I am proposing that the Region's review of the Phase I RFI be completed by June 30, 1998. Could you please let Paul Little (x6-4460) or I (x6-6199) know ASAP whether you have any problems with this proposal? Thank you.

cc: Little  
File



**FEDEX NUMBER:** 1006-8942-1

December 4, 1997

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (DRE-9J)  
77 West Jackson Street  
Chicago, Illinois 60604

Subject: Submittal of Draft Phase I RCRA Facility Investigation Report  
RCRA Facility Investigation  
Docket No.: V-W-011-94  
BASF Corporation, Wyandotte, Michigan

Dear Ms. Sharrow:

BASF Corporation is submitting three copies of the Draft Phase I RCRA Facility Investigation Report for the Wyandotte Facility in accordance with the time extension granted in your letter dated November 3, 1997.

The report includes:

- the report proper
- Appendix A -- Excerpts of Geological Data and Analytical Results from Prior Investigations;
- Appendix B -- Soil Boring and Monitoring Well Logs;
- Appendix C -- Aquifer Testing Data and Analyses;
- Appendix D -- Data Validation Reports Prepared by Environmental Standards Inc.;
- Appendix E -- Field Parameter and Groundwater Elevation Summary Tables;
- Appendix F -- Exposure Assumptions for Chemical Intake Estimates;
- Appendix G -- Exposure and Risk Calculations; and
- Appendix H -- GTI TRIP Report

The information in Appendix D is quite extensive and is significantly consolidated. The consolidated version will be sent to MDEQ and to the Bacon Memorial Public Library in Wyandotte.

Based upon information obtained from the groundwater extraction evaluation conducted during the Phase I activities, it appears that the clay and peat unit is an aquitard and that there are probably two water-bearing zones. The first zone lies within the fill, and the second zone consists of the native sand unit where the groundwater extraction wells are screened. In addition, data indicate that groundwater is leaving the Facility. Based upon the information gathered to date, the volume of groundwater leaving the Facility cannot be quantified, and it has not been established whether the groundwater is "contaminated" as defined in the 1986 Consent Order. BASF will be gathering additional information during the up coming months. As you and Mr. Roberts have briefly discussed, we believe that it will be advantageous to have an informal meeting in January to discuss the report.

I certify that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to evaluate the information submitted. I certify that the information contained in or accompanying this submittal is true, accurate, and complete. As to those identified portion(s) of this submittal for which I cannot personally verify the accuracy, I certify that this submittal and all attachments were prepared in accordance with procedures designed to assure that qualified personnel properly gathered and evaluated the information submitted. Based on my inquiry of the person or persons who manage the system, or those directly responsible for gathering the information, or the immediate supervisor of such person(s), the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

Should you need additional information, please contact us at your convenience.

Sincerely,

*Don Yarborough*  
Don Yarborough

Don Yarborough  
Wyandotte Site Manager

bdr

Attachment - Draft Phase I RCRA Facility Investigation Report and appendices

cc: B. Roberts - BASF  
D. Marian - QST  
R. Blayer - MDEQ Lansing  
L. Aubuchon - MDEQ Livonia  
J. Russell - MDEQ Livonia  
A. Danford - Quanterra (letter only)  
B. Wallace - Bacon Memorial Public Library

**From:** John Koehnen <JGK@techlawinc.com>  
**To:** R5WST.R5RCRA(SHARROW-DIANE)  
**Date:** 2/6/98 11:44am  
**Subject:** BASF

**\*\* High Priority \*\***

Diane:

As a follow up to my recent voice mail message, just a quick note to update you on the status of BASF. The reviews are underway and are proceeding as planned. However, as per my voice mail message, the review of the geology of the site has identified some data gaps which we need to fill in to get an accurate picture of the site conditions. If you have or can acquire the following pieces of information it would be appreciated. Alternatively, if you wish, I can contact the QST Project Manager and request this information.

The following items are outstanding:

- Several wells are depicted on the geologic cross sections, but for several of the locations (e.g., PE13NB, PE10NB, P39N, DNR4, to name only a few), the corresponding well logs are not included for review. These data points may relate to piezometer installations, but logs should still be available.
- A Groundwater Profile is not included but would be valuable to better define the interrelationship between the site topography, the geologic/fill conditions and the movement of GW within the system.
- The table of water levels only includes those wells which were used to develop GW contour elevations. Data of this type for all, or most, of the wells at the site is important to better define the site hydrogeology and to evaluate the cross sections.
- In addition, water levels, well ID and locations are needed for the observation wells evaluated during the pump tests at the site. The water level information for these wells should relate to the time period when the pump tests were performed.

If you have any questions regarding the information needs, please contact me at your convenience (312-345-8938)

John Koehnen

**CC:** R5CHG.IN("SPhillips@techlawinc.com","PBrown-Deroch...

3 November 1997

DRE-9J COPY

Bruce Roberts  
Project Manager  
BASF Corporation  
1609 Biddle Avenue  
Wyandotte, Michigan 48192

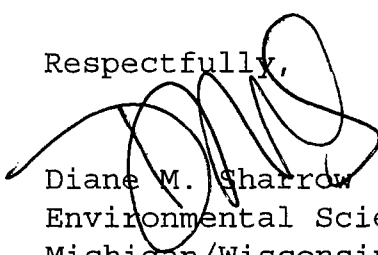
RE: BASF, Inc. (North Works)  
U.S. EPA ID. NO. : MID 064 197 742

Dear Mr. Roberts:

In response to your letter of 28 October 1997, I am approving an extension of time to submit the RFI Report. I realize that the large volume of data to be validated is due in part to the U.S. EPA's requirement for full Appendix IX sampling, as well as the discovery of a third Prussian Blue area. The RFI Report is now due December 5, 1997. The U.S. EPA expects to finish its review of the RFI Report in approximately six months; June 30, 1998. It is my hope to approve or conditionally approve the RFI report by that date.

If you have any questions or concerns, please contact me at (312) 886-6199. Please note that my facsimile number is now (312) 353-4342 and my Mailcode is now DRE-9J.

Respectfully,



Diane M. Sharrow  
Environmental Scientist  
Michigan/Wisconsin section  
Enforcement and Compliance Assurance Branch  
Waste, Pesticides and Toxics Division

cc: R. Blayer, MDEQ-Lansing  
L. Aubuchon, MDEQ - Livonia  
J. Russell, MDEQ - Livonia

-2-

bcc: Paul Little  
Gerry Phillips

BASF Corporation

**BASF**

October 28, 1997

RECEIVED  
NOV 03 1997

CERTIFIED MAIL - RETURN RECEIPT REQUESTED

P 607 933 816

DIVISION FRONT OFFICE  
Waste, Pesticides & Toxics Division  
U.S. EPA - REGION 5

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (DRE-8J)  
77 West Jackson Street  
Chicago, Illinois 60604

Subject: Submission of draft RFI report  
BASF Corporation  
USEPA ID Number MID064197742  
Wyandotte, Michigan

Dear Ms. Sharrow:

Per our telephone conversation yesterday, BASF Corporation is requesting a delay until December 5th for submitting the draft RFI report to USEPA.

It took longer than expected for Environmental Standards to complete the data validation. BASF did not receive the last originally scheduled validated package until the latter part of September. The validated data for the confirmation sampling for the third Prussian Blue area was not received until early October.

There is a massive amount of data to examine (almost 70,000 records including laboratory checks) and it has taken a substantial effort to re-arrange the data by AOCs, SWMUs, and wells and to perform the necessary statistical manipulations.

If you need additional information, please let me know.

Thank you.

Sincerely,

*Bruce Roberts*

Bruce Roberts  
Project Manager

cc: D. Yarborough - BASF  
D. Marian - QST  
R. Blayer - MDEQ Lansing  
L. Aubuchon - MDEQ Livonia  
J. Russell - MDEQ Livonia  
B. Wallace - Bacon Memorial Public Library

September 22, 1997

BASF Corporation  
Wyandotte RFI

The following decision tree will be followed when the data validation reports contain both the initial analytical results and re-test analytical results.

1. This situation is caused by matrix interferences. The surrogate recoveries were outside the acceptable criteria, the sample was re-extracted and re-analyzed, the surrogate recoveries were still outside the acceptable criteria, the holding time was exceeded -- use the initial results.
2. This situation is caused by matrix interferences. The surrogate recoveries were outside the acceptable criteria, the sample was re-extracted and re-analyzed, the surrogate recoveries were still outside the acceptable criteria, the holding time was not exceeded -- use the initial results.
3. The surrogate recoveries were outside the acceptable criteria, the sample was re-extracted and re-analyzed, the surrogate recoveries were within the acceptable criteria, the holding time was exceeded -- use the larger results to be conservative.
4. The method blank and/or laboratory control sample were outside the acceptable criteria, the sample was re-extracted and re-analyzed, the method blank and/or laboratory control sample were within the acceptable criteria, the holding time was exceeded -- use the re-extracted results.

RETESTS  
BASF CORPORATION  
WYANDOTTE RFI

Sample_Delivery_Group	CLIENT_ID	Fraction	Method_Name	MATRIX	ReTestCode	Analysis Used
A6G170130	SG001RFIMW24	SVOA	8270B	SOLID	X1	Initial
A6G250145	SG010RFIMW07	SVOA	8270B	SOLID	X1	Initial
A6G250145	SG013RFIMW07	SVOA	8270B	SOLID	X1	Initial
A6H020118	SG006SWMUFSP11	SVOA	8270B	SOLID	X1	Initial
A6I100127	SW000AOC6	SVOA	8270B	WATER	X1	Larger - initial
A6I120184	SG014SWMUHSP02C	HERB	8150B	SOLID	X1	Larger - initial
A6I190152	MW000RFIMW-11	SVOA	8270B	WATER	X1	Larger - initial
A6J010134	MW000PM3NB	SVOA	8270B	WATER	X1	Initial
A6J010134	MW000RFIMW-18 (MS/MSD)	SVOA	8270B	WATER	X1	Initial
A6J010134	MW000RFIMW-23 (MS/MSD)	SVOA	8270B	WATER	X1	Initial
A6J300159	MW000RFIMW-28	SVOA	8270B	WATER	X1	Initial
A6J300159	MW000RFIMW-28D	SVOA	8270B	WATER	X1	Initial
A6L190128	MW000P-34-N	SVOA	8270B	WATER	X1	Re-analysis
A6L190128	MW000PMINA	SVOA	8270B	WATER	X1	Re-analysis
A6L190128	MW000RFIMW-10 (MS/MSD)	SVOA	8270B	WATER	X1	Initial
A6L190128	MW000RFIMW-11	SVOA	8270B	WATER	X1	Larger - reanalysis
A6L190128	MW000RFIMW-24 (MS/MSD)	SVOA	8270B	WATER	X1	Re-analysis
A6L190128	MW000RFIMW-25	SVOA	8270B	WATER	X1	Re-analysis
A6L190128	MW000RFIMW-25D	SVOA	8270B	WATER	X1	Re-analysis
A6L190128	MW000RFIMW-26	SVOA	8270B	WATER	X1	Re-analysis
A6L190128	MW000RFIMW-27	SVOA	8270B	WATER	X1	Re-analysis
A6L190128	MW000RFIMW-29	SVOA	8270B	WATER	X1	Re-analysis
A6L190128	MW000RFIMW-3	SVOA	8270B	WATER	X1	Initial
A6L190128	MW000RFIMW-5	SVOA	8270B	WATER	X1	Larger - reanalysis
A6L190128	MW000RFIMW-6	SVOA	8270B	WATER	X1	Re-analysis
A6L190128	MW000RFIMW-7	SVOA	8270B	WATER	X1	Re-analysis
A6L190128	MW000RFIMW-8	SVOA	8270B	WATER	X1	Re-analysis
A6L190128	MW000RFIMW-9	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000MWPMINA	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000P-34-N	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-1	SVOA	8270B	WATER	I1	Re-analysis
A7C190143	MW000RFIMW-10	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-11	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-12	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-2	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-22	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-23	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-24	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-25	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-26	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-27	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-28	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-29	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-3	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-30	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-4	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-5	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-6	SVOA	8270B	WATER	X1	Re-analysis
A7C190143	MW000RFIMW-7	SVOA	8270B	WATER	X1	Re-analysis
A7F030144	MW000RFIMW-01	SVOA	8270B	WATER	I1	Re-analysis
A7F030144	MW000RFIMW-23	SVOA	8270B	WATER	X1	Larger - initial
A7F030144	MW000RFIMW-24	SVOA	8270B	WATER	X1	Larger - reanalysis
A7F030144	MW000RFIMW-25 (MS/MSD)	SVOA	8270B	WATER	X1	Larger - initial
A7F030144	MW000RFIMW-25 (MS/MSD)	VOA	8260A	WATER	X1	Initial
A7F030144	MW000RFIMW-30	SVOA	8270B	WATER	I1	Re-analysis



**Facsimile  
Transmittal**

*Quanterra Incorporated  
4101 Shuffel Drive NW  
North Canton, Ohio 44720*

*(216) 966-9785 Telephone  
(216) 497-0772 Fax*



Date: Friday, July 12, 1996

Deliver to: Dave Payne

Company & US EPA

Location: Chicago, IL

Fax Destination: (312) 353-4342

From: Tom Himes

Note: This telecopy contains 3 page(s) which includes this cover page. Please call the number at the top of this form if you do not receive all pages.

Comments:

Dave:

Please see the following addendum to our Cyanide SOP as drawn up by Mark Bruce. Please contact myself or Mark with questions or concerns.

Thanks,

Tom

**Confidentiality Notice:**

The documents accompanying this telecopy transmission contain confidential information which is legally privileged. The information is intended only for the use of the recipient named above. If you received this telecopy in error, please notify us immediately by telephone to arrange for the return of the documents to us, and you are hereby notified that any disclosure, copying, distribution, or the taking of any action in reliance on the contents of this telecopied information is strictly prohibited.

BASF RFI  
SOP Addendum  
NC-WC-0031 & NC-WC-0032  
Total Cyanide 9012

The following amendments will be made to the above referenced SOPs. These amendments are based on the comments from US EPA upon review of site requirements and laboratory procedures.

Addendum for NC-WC-0032 for all samples.

change 7.2.1 Change 2.51 g of potassium cyanide to 2.11g potassium ferricyanide.

change 9.4 The matrix spike/duplicate frequency has been increased from one per batch to one per 7 samples. Consult with the project manger for specific samples to be spiked. The goal is to spike each sampling area at the site at least once.

Addendum for NC-WC-0032 for Prussian blue area samples.

add 7.1.22. Sodium thiocyanate : reagent grade. Prepare a spiking solution at a concentration 100X greater than the measured cyanide for the appropriate Prussian blue area samples.

add 7.1.23. Prussian blue sample digestion fluid/absorber solution: combine 10 g NaOH, 1 g CdCO<sub>3</sub>, in a 1 L volumetric flask. Dissolve the solids in reagent water and dilute to volume.

add 9.5. Three Prussian blue area basic sample digestates will be spiked with 1 mL of the sodium thiocyanate solution from 7.1.22. The increase (if any) in measured cyanide concentration relative to the original sample shall be reported as "percentage increase". Use the following equation:

$$\%CN_{increase} = \frac{Conc_{SCN\_spike} - Conc_{unspiked}}{Conc_{unspiked}} \times 100$$

where  $Conc_{SCN\_spike}$  is the cyanide concentration measured in the thiocyanate spiked sample  
 $Conc_{unspiked}$  is the cyanide concentration measured in the unspiked sample

add 11.2.1.4. Prussian blue cyanide preparation procedure: Combine 1 g of air dried and homogenized sample with 50 mL Prussian blue sample digestion fluid (7.1.23) in 120 mL snap seal container. Place in a boiling water bath for 3 hours. Check to confirm that pH>11. If not add solid NaOH to raise pH>12 and reheat on water bath for 3 hours.

change 11.2.4.1 Change 1.0g sample and 50 mL reagent water to the entire contents (50 mL) of the snap seal container used in the Prussian blue digestion (11.2.1.4). Change absorber solution to the Prussian blue absorber solution (7.1.23). Add 1 mL of SCN spike solution if appropriate. Also, spike SCN into a separate reagent water blank. Note: initial sample color on benchsheet. Add a piece of appropriate pH paper to be monitored when adjusting pH in next step.

add to 11.2.4.3 Be sure to wait 3 minutes between adding the sulfuric acid and the magnesium chloride solution. Make sure the pH is <2.

add to 11.2.4.5 Note absorber solution color on benchsheet. Test absorber solution pH. Note if not >12 and notify supervisor. Test absorber solution with lead acetate paper. Treat with  $\text{CdCO}_3$  if sulfide is detected. Shake and retest. If volume has increased to more than 52 mL, note final volume for use in final calculations. Allow precipitate to settle before withdrawing aliquot for analysis. Centrifuge if necessary in order to produce a particulate free aliquot.

**BASF RFI  
SOP Addendum  
LM-WALN-4110/CORP-GC-0001  
Herbicide 8150A**

The following amendments will be made to the above referenced SOPs. These amendments are based on the comments from US EPA upon review of site requirements and laboratory procedures.

The Herbicide preparation and analyses will be modified as follows:

**Organic Extraction - Solid Matrix:**

- 5 grams of soil will be used instead of 50 grams of soil as the initial weight.
- Samples will be surrogated with 2ug/ml of DCAA.
- The spiking solution used for waters will be used for Laboratory Check (LCS) and MS/MSD.
- Samples will be taken to 10 ml final volume.

**Analysis:**

- Both the solid and water extracts will be analyzed without a dilution unless matrix interference is found.

**BASF RFI  
SOP Addendum  
CORP-MT-0001**

The following information serves as an addendum to the Quanterra SOP for metals analysis by 6010A. The modifications to this SOP are based on comments from US EPA in reference to the BASF RFI QAPjP.

The following modifications will be made to this SOP in order to achieve the Targeted Quantitation Limits that are listed on Table 7-4 of the QAPjP:

**Table IV. ICP Calibration and Calibration Verification Standards**

- The following CRI concentrations have been modified to reflect project reporting limits for ICP:

<u>Element</u>	<u>CRI (ug/L)</u>	<u>Reporting Limit (ug/L)</u>
Barium	20	10
Copper	20	10

**Table IVA. Trace Calibration and Calibration Verification Standards**

- The following CRI concentrations have been modified to reflect project reporting limits for Trace ICP:

<u>Element</u>	<u>CRI (ug/L)</u>	<u>Reporting Limit (ug/L)</u>
Antimony	10	5.0
Arsenic	10	5.0
Cadmium	2.0	1.0

Revisions to tables are attached.

**INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION  
SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE  
ELEMENT ANALYSIS, METHOD 6010A AND METHOD 200.7  
APPENDIX A - TABLES**

**SOP No. CORP-MT-0001  
Revision No. 1  
Revision Date: 7-21-95  
Page: 34 of 51**

**TABLE IV. ICP Calibration and Calibration Verification Standards**

Element	Calibration Level	RL (ug/L)	CRI (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	400	25000	50000
Antimony	10000	60	120	1000	5000
Arsenic	10000	300	600	1000	5000
Barium	10000	200	20	1000	5000
Beryllium	10000	5	10	1000	5000
Cadmium	10000	5	10	1000	5000
Calcium	100000	5000	10000	25000	50000
Chromium	10000	10	20	1000	5000
Cobalt	10000	50	100	1000	5000
Copper	10000	25	20	1000	5000
Iron	100000	100	100	25000	50000
Lead	10000	100	200	1000	5000
Lithium	10000	50	100	1000	5000
Magnesium	100000	5000	10000	25000	50000
Manganese	10000	15	20	1000	5000
Molybdenum	10000	40	80	1000	5000
Nickel	10000	40	80	1000	5000
Phosphorous	10000	300	600	1000	5000
Potassium	100000	5000	10000	25000	50000
Selenium	10000	250	500	1000	5000
Silver	2000	10	20	500	1000
Sodium	100000	5000	10000	25000	50000
Strontium	10000	50	100	1000	5000
Thallium	20000	2000	4000	5000	10000
Vanadium	10000	50	100	1000	5000
Zinc	10000	20	40	1000	5000
Boron	10000	200	400	1000	5000
Silicon	10000	500	1000	1000	5000
Tin	10000	100	200	1000	5000
Titanium	10000	50	100	1000	5000
Bismuth	10000	200	400	1000	5000
Zirconium	10000	100	200	1000	5000
Tellurium	10000	500	1000	1000	5000
Thorium	10000	500	1000	1000	5000
Uranium	10000	500	1000	1000	5000
Tungsten	10000	500	1000	1000	5000
Palladium	10000	100	200	1000	5000

**INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION  
SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE  
ELEMENT ANALYSIS, METHOD 6010A AND METHOD 200.7  
APPENDIX A - TABLES**

**SOP No. CORP-MT-0001  
Revision No. 1  
Revision Date: 7-21-95  
Page: 35 of 51**

**TABLE IVA. Trace Calibration and Calibration Verification Standards**

Element	Calibration Level	RL (ug/L)	CRI (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	100	12500	25000
Antimony	1000	10	10	250	500
Arsenic	1000	10	10	250	500
Barium	4000	10	20	1000	2000
Beryllium	4000	5	10	1000	2000
Cadmium	1000	2	2	250	500
Calcium	100000	5000	10000	25000	50000
Chromium	4000	5	10	1000	2000
Cobalt	4000	50	40	1000	2000
Copper	4000	25	50	1000	2000
Iron	50000	100	100	12500	25000
Lead	1000	3	6	250	500
Magnesium	100000	5000	10000	25000	50000
Manganese	4000	15	20	1000	2000
Molybdenum	4000	40	20	1000	2000
Nickel	4000	40	80	1000	2000
Potassium	100000	5000	10000	25000	50000
Selenium	1000	5	10	250	500
Silver	2000	5	10	500	1000
Sodium	100000	5000	10000	25000	50000
Thallium	2000	10	20	500	1000
Vanadium	4000	50	40	1000	2000
Zinc	4000	20	40	1000	2000

**Facsimile  
Transmittal**

*Quanterra Incorporated  
4101 Shuffel Drive NW  
North Canton, Ohio 44720*

*(216) 966-9785 Telephone  
(216) 497-0772 Fax*



Date: Friday, August 02, 1996

Deliver to: Dave Payne

Company & U.S. EPA

Location:

Fax Destination: (312) 353-4342

From: Tom Himes

Note: This telecopy contains 2 page(s) which includes this cover page. Please call the number at the top of this form if you do not receive all pages.

Comments:

Dave:

Please review the attached Cyanide addendum which has been revised per our recent discussion. If any additional changes are necessary, please contact me at (330)966-9785.

Thanks,

Tom Himes

c: Bruce Roberts, BASF

**Confidentiality Notice:**

The documents accompanying this telecopy transmission contain confidential information which is legally privileged. The information is intended only for the use of the recipient named above. If you received this telecopy in error, please notify us immediately by telephone to arrange for the return of the documents to us, and you are hereby notified that any disclosure, copying, distribution, or the taking of any action in reliance on the contents of this telecopied information is strictly prohibited.



BASF RFI  
SOP Addendum  
NC-WC-0031 & NC-WC-0032  
Total Cyanide 9012

The following amendments will be made to the above referenced SOPs. These amendments are based on the comments from US EPA upon review of site requirements and laboratory procedures.

Addendum for NC-WC-0032 for all samples.

change 7.2.1 Change 2.51 g of potassium cyanide to 2.11g potassium ferricyanide.

change 9.4 The matrix spike/duplicate frequency has been increased from one per batch to one per 7 samples. Consult with the project manger for specific samples to be spiked. The goal is to spike each sampling area at the site at least once.

Addendum for NC-WC-0032 for Prussian blue area samples.

add 7.1.22. Sodium thiocyanate : reagent grade. Prepare a spiking solution at a concentration 100X greater than the measured cyanide for the appropriate Prussian blue area samples.

add 9.5. Three Prussian blue area samples will be spiked with 1 mL of the sodium thiocyanate solution from 7.1.22 in order to assess the potential impact of thiocyanate in the original samples on the measured cyanide concentration. These matrix spikes do not replace the cyanide matrix spikes mentioned above (9.4). The increase (if any) in measured cyanide concentration relative to the original sample shall be reported as "percentage increase". Use the following equation:

$$\%CN_{increase} = \frac{Conc_{SCN\_spike} - Conc_{unspiked}}{Conc_{unspiked}} \times 100$$

where  $Conc_{SCN\_spike}$  is the cyanide concentration measured in the thiocyanate spiked sample  
 $Conc_{unspiked}$  is the cyanide concentration measured in the unspiked sample

Note: 11.3.2.1 of NC-WC-0031 already addresses sulfide testing and precipitation.

BASF Corporation

**BASF**

May 23, 1996

**RECEIVED**  
MAY 24 1996

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (DRE-8J)  
77 West Jackson Street  
Chicago, Illinois 60604

**OFFICE OF RCRA  
WASTE MANAGEMENT DIVISION  
EPA, REGION V**

RE: Additional RFI items


Dear Ms. Sharrow:

Enclosed is the original signature sheet that has been signed by everyone except for you and Willie Harris. Please complete the signature and date portion and return the page to me for distribution.

Enclosed is a copy of the revised project schedule. It is contingent upon a rapid resolution of the remaining cyanide issue.

Also enclosed is ESI's response to Mr. Payne's comments concerning the data validation that were attached to your April 23 letter.

Sincerely yours,

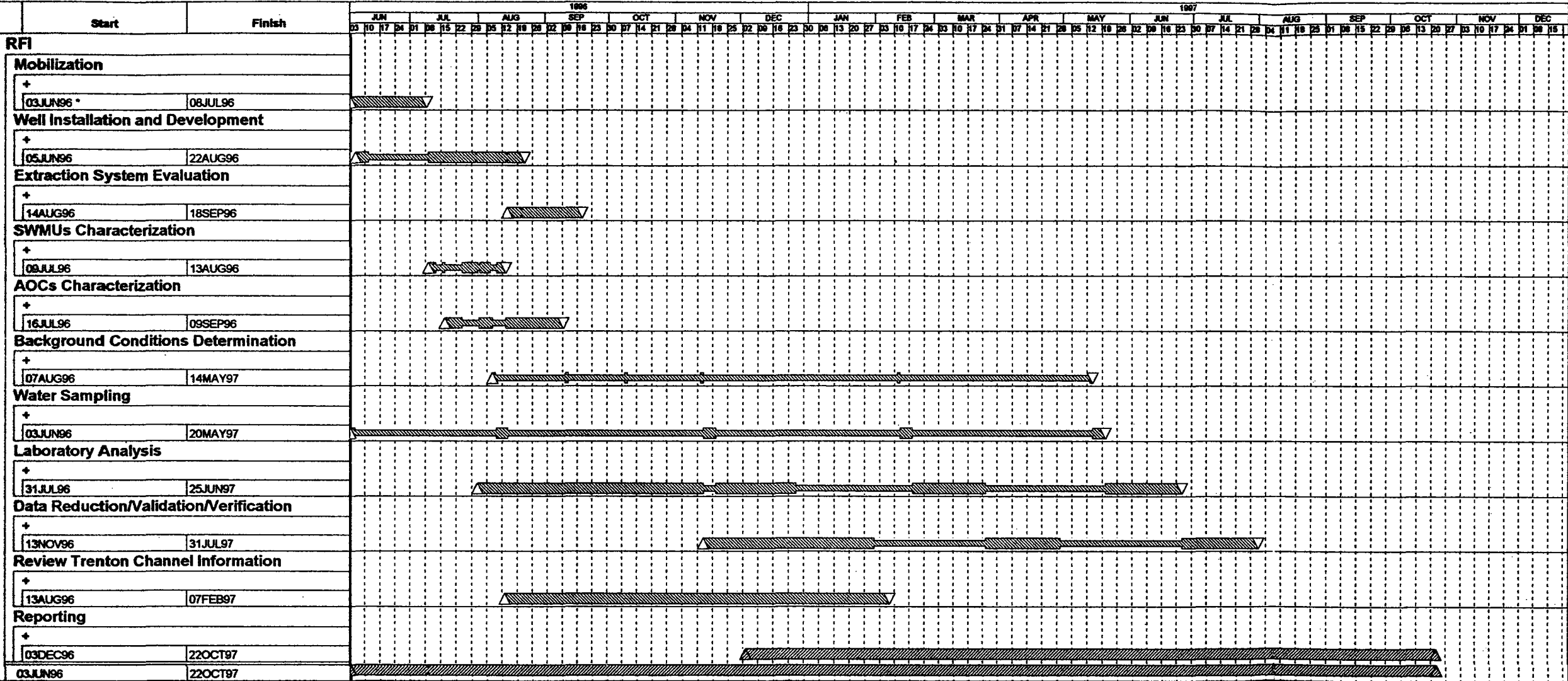
  
Bruce Roberts  
Project Coordinator

Enclosures

z:\winword\05236.ltr

cc w/o signature page:

D. Yarborough - BASF  
R. Veenstra - ESE  
R. Vitale - ESI  
R. Blayer - MDEQ Lansing  
L. Aubuchon - MDEQ Livonia  
J. Russell - MDEQ Livonia  
T. Himes - Quanterra



Environmental  
Science &  
Engineering, Inc.

Data date	03JUN96	Date	Revision	Checked	Approved
Start date	03JUN96	20MAY96	1.0 (Draft)	DMB	RBV
Finish date	22OCT97	21MAY96	2.0 (Draft)	DMB	RBV
		21MAY96	3.0 (Final)	RBV	BDR
© Primavera Systems, Inc.					

Thomas M. Himes  
Project Manager



Quanterra Incorporated  
4101 Shuffel Drive, NW  
North Canton, Ohio 44720

330 497-9396 Telephone  
330 497-0772 Fax

May 23, 1996

Ms. Diane Sharrow  
United States Environmental Protection Agency  
Region V, (DRE-8J)  
77 West Jackson Street  
Chicago, IL 60604



Re: BASF Corporation RCRA Facility Investigation / ICP Trace Technology

Dear Ms. Sharrow:

As requested by Mr. Dave Payne, please see the enclosed data for Standard Reference Material (SRM) analyses. This data has been generated in support of the BASF RFI that is scheduled to be conducted in Wyandotte, MI.

The analyses of the SRM samples was requested by Mr. Payne in an effort to prove the capabilities of method SW 846 6010A - ICP Trace. This is a relatively new technology that allows the laboratory to achieve a lower range of detection for selected elements that would typically be analyzed by a graphite furnace (GFAA).

The benefits of the ICP Trace include reduced analysis time, a wider range of linearity, and reduced costs. This technology has been used for the past three years, and has proven to be a very reliable method of analysis.

Please contact myself, or Susan Palmer of our metals laboratory at (330) 497-9396 for a full explanation of the attached data.

Sincerely,

A handwritten signature in cursive script that reads "Thomas M. Himes".

Thomas M. Himes

Enclosures

cc: Bruce Roberts, BASF Corporation  
Susan Palmer, Quanterra Environmental Services

## EXPANDED DELIVERABLE DATA REVIEW SHEET

LOT #

SRM's for BASF (David Payne)

Analyzed by  
TJALIE TRACE  
ICP

Fraction: VOA

BNA

PEST/PCB

METALS

WET CHEM

(CIRCLE ONE)

Date Reviewed:

5-15-96

Reviewed By:

Susan Palmer

QA/QC Comments (Please note if no problems encountered):

SRM 2709 was diluted 1:2 due to Y present  
in the sample at such a level as to elevate the raw Y counts by 7%.

SRM 2710 was diluted 1:10 due to the presence  
of Mn at a level above our instrument  
linearity. Mn affects a number of elements.

SRM 2711 was diluted 1:5 due to Y present in  
the sample at such a level as to elevate  
the raw Y counts by >30%.

☐

Form VI represents the duplication of the actual MS/MSD results.

☐

No Form VIII included - No MSA's performed.

☐

No Form IX included - No serial dilution performed.

☐

No Form V or Form VI provided - No MS/MSD performed.

☐

No Form IX provided for this lot. The serial dilution was performed on a sample in lot \_\_\_\_\_ from this client and from the same QC batch as the samples in this lot.

Level IIC Review:

Calculations:

SP

Units:

Prep/Anal. Date:

J

Level I done:

J

Level IIa done:

SP

5-16-96

Susan Palmer

● INTSTD

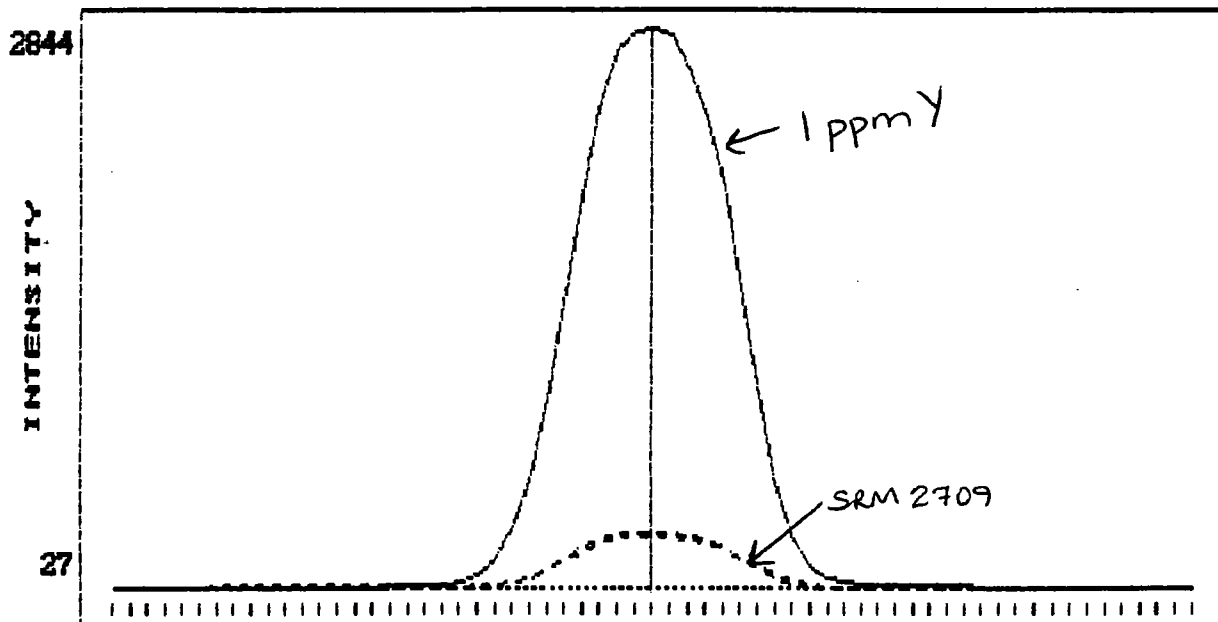
INT

SN: Y 1PPM

05/15/96 11:39:00

Y 371.030 Intensity = 2844 SCALE: X 1.000

Maximum(s): 2844 27 296 307



Cursor Wavelength: 371.030

Spec Pos: 0

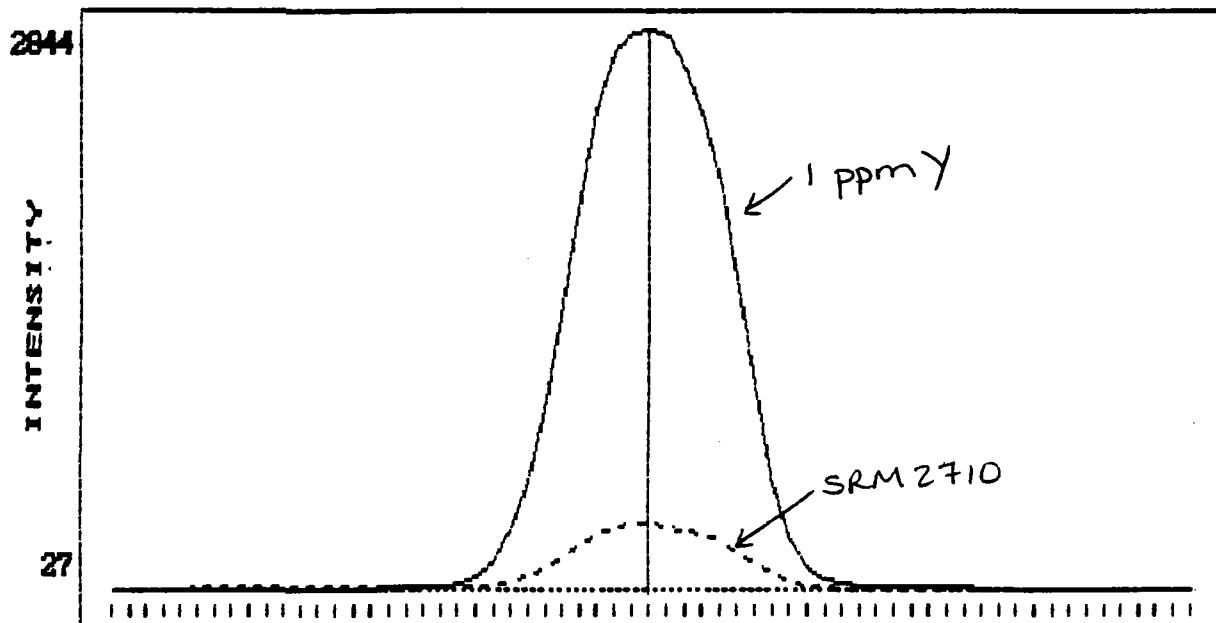
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SRM2709 291

Y100PPB 304

INTSTD INT SN: Y 1PPM  
Y 371.030 Intensity = 2844 SCALE: X 1.000  
Maximum(s): 2844 27 347

05/15/96 11:39:00



Cursor Wavelength: 371.030

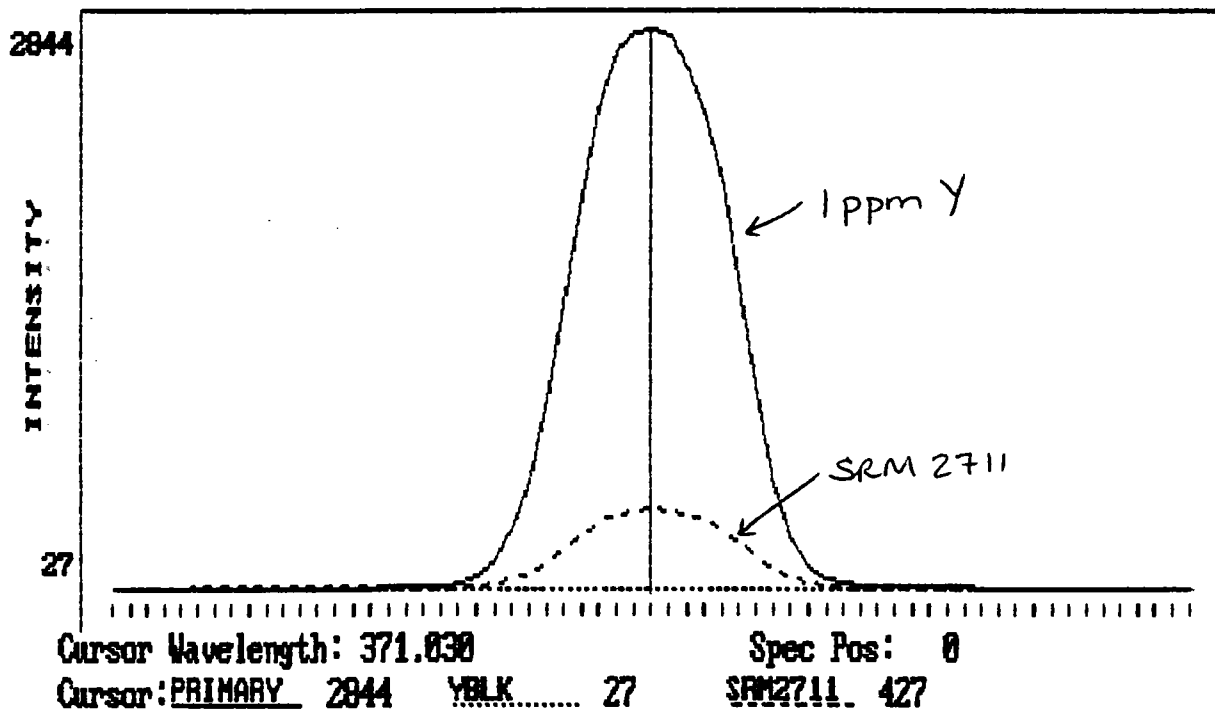
Spec Pos: 0

Cursor: PRIMARY 2844 YBLK..... 27

SRM2710. 347

POINT STD INT SN: Y 1PPM  
Y 371.030 Intensity = 2844 SCALE: X 1.000  
Maximum(s): 2844 27 427

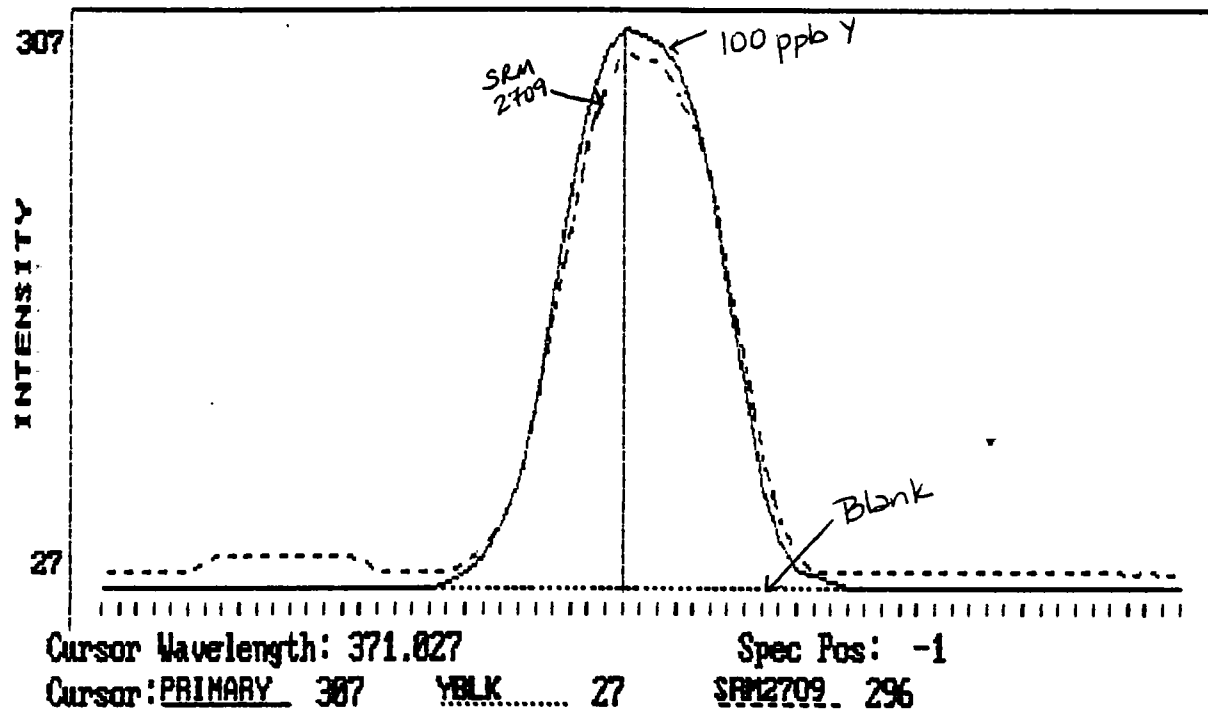
05/15/96 11:39:00





● INTSTD INT SN: Y 100PPB  
Y 371.030 Intensity = 304 SCALE: X 1.000  
Maximum(s): 307 27 296

05/15/96 11:52:13



● INTSTD

INT

SN: Y 100PPB

05/15/96 11:52:13

Y 371.030

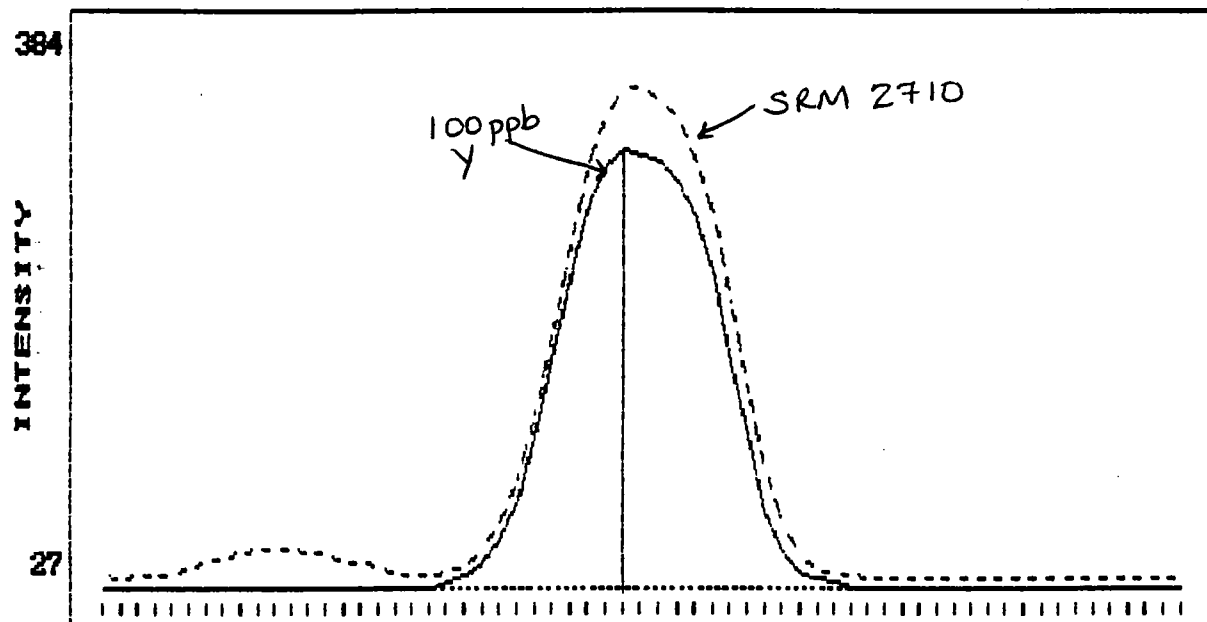
Intensity = 384

SCALE: X .800

Maximum(s): 387

27

347



Cursor Wavelength: 371.027

Spec Pos: -1

Cursor: PRIMARY 387

YBLK..... 27

SRM2710. 347

POINT STD

INT

SN: Y 100PPB

05/15/96 11:52:13

Y 371.030

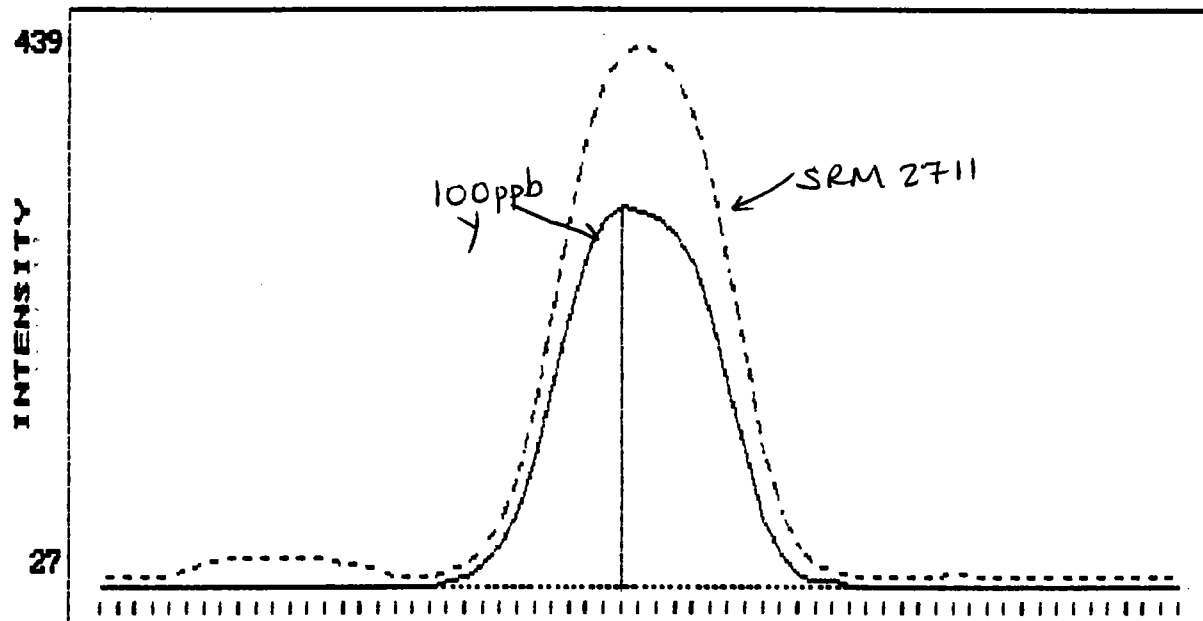
Intensity = 304

SCALE: X .700

Maximum(s): 307

27

427



Cursor Wavelength: 371.027

Spec Pos: -1

Cursor: PRIMARY 307 YBLK..... 27

SRM2711. 419



LABORATORY CONTROL SAMPLE

Client:

SDG No.: SRM

Aqueous LCS Source:

[illegible]

7.  
LABORATORY CONTROL SAMPLE

Client:

SDG No.: SRM

Aqueous LCS Source: \_\_\_\_\_

FORM VII - IN

7  
LABORATORY CONTROL SAMPLE

Client: \_\_\_\_\_

SDG No. : SRM

Aqueous LCS Source: \_\_\_\_\_

[illegible]

7  
LABORATORY CONTROL SAMPLE

Client:

SDG No.: SRM

Aqueous LCS Source: \_\_\_\_\_

FORM VII - IN



7  
LABORATORY CONTROL SAMPLE

Client: \_\_\_\_\_

SDG No. : SRM

Solid LCS Source: 2710

Aqueous LCS Source:

[illegible]

7  
LABORATORY CONTROL SAMPLE

Client:

SDG No.: SRM

Aqueous LCS Source:

[illegible]

7  
LABORATORY CONTROL SAMPLE

Client: \_\_\_\_\_

SDG No.: SRM

Aqueous LCS Source: \_\_\_\_\_

[illegible]

7  
LABORATORY CONTROL SAMPLE

Client: \_\_\_\_\_

SDG No.: SRM\_\_\_\_\_

Aqueous LCS Source: \_\_\_\_\_

[illegible]



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material 2709

#### San Joaquin Soil

#### Baseline Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. SRM 2709 is an agricultural soil that was oven-dried, sieved, and blended to achieve a high degree of homogeneity. A unit of SRM 2709 consists of 50 g of the dried material.

The certified elements for SRM 2709 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should not be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

#### NOTICE AND WARNINGS TO USERS

**Expiration of Certification:** This certification is valid for 5 years from the date of shipment from NIST. Should any of the certified values change before the expiration of the certification, purchasers will be notified by NIST. Return of the attached registration card will facilitate notification.

**Stability:** This material is considered to be stable; however, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser.

**Use:** A minimum sample weight of 250 mg (dry weight - see Instructions for Drying) should be used for analytical determinations to be related to the certified values on this Certificate of Analysis.

To obtain the certified values, sample preparation procedures should be designed to effect complete dissolution. If volatile elements (i.e., Hg, As, Se) are to be determined, precautions should be taken in the dissolution of SRM 2709 to avoid volatilization losses.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The overall direction and coordination of the analyses were under the chairmanship of M.S. Epstein and R.L. Watters, Jr., of the NIST Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills and J.S. Kane.

Gaithersburg, MD 20899  
August 23, 1993  
(Revision of certificate dated 10-30-92)

Thomas E. Gills, Acting Chief  
Standard Reference Materials Program

(over)

**Instructions for Drying:** When nonvolatile elements are to be determined, samples should be dried for 2 h at 110 °C. Volatile elements (i.e., Hg, As, Se) should be determined on samples as received; separate samples should be dried as previously described to obtain a correction factor for moisture. Correction for moisture is to be made to the data for volatile elements before comparing to the certified values. This procedure ensures that these elements are not lost during drying. The weight loss on drying has been found to be in the range of 1.8 to 2.5%.

**Source and Preparation of Material:** The U.S. Geological Survey (USGS), under contract to the NIST, collected and processed the material for SRM 2709. The soil was collected from a plowed field, in the central California San Joaquin Valley, at Longitude 120° 15' and Latitude 36° 30'. The collection site is in the Panoche fan between the Panoche and Cantu creek beds. The top 7.5-13 cm (3-5 in) of soil containing sticks and plant debris was removed, and the soil was collected from the 13 cm level down to a depth of 46 cm (18 in) below the original surface. The material was shoveled into 0.114 m<sup>3</sup> (30 gal) plastic buckets and shipped to the USGS laboratory for processing.

The material was spread on 30.5 cm x 61 cm (1 ft x 2 ft) polyethylene-lined drying trays in an air drying oven and dried for three days at room temperature. The material was then passed over a vibrating 2 mm screen to remove plant material, rocks, and large chunks of aggregated soil. Material remaining on the screen was deaggregated and rescreened. The combined material passing the screen was ground in a ball mill to pass a 74 µm screen and blended for 24 h. Twenty grab samples were taken and measured for the major oxides using x-ray fluorescence spectrometry and for several trace elements using inductively coupled plasma atomic emission analysis to provide preliminary assessment of the homogeneity of the material prior to bottling. The material was bottled into 50 g units and randomly selected bottles were taken for the final homogeneity testing.

**Analysis:** The homogeneity, using selected elements in the bottled material as indicators, was assessed using x-ray fluorescence spectrometry and neutron activation analysis. In a few cases, statistically significant differences were observed, and the variance due to material inhomogeneity is included in the overall uncertainties of the certified values. The estimated relative standard deviation for material inhomogeneity is less than 1% for those elements for which homogeneity was assessed.

**Certified Values and Uncertainties:** The certified values are weighted means of results from two or more independent analytical methods, or the mean of results from a single definitive method, except for mercury. Mercury certification is based on cold vapor atomic absorption spectrometry used by two different laboratories employing different methods of sample preparation prior to measurement. The weights for the weighted means were computed according to the iterative procedure of Paule and Mandel (NBS Journal of Research 87, 1982, pp. 377-385). The stated uncertainty includes allowances for measurement imprecision, material variability, and differences among analytical methods. Each uncertainty is the sum of the half-width of a 95% prediction interval and includes an allowance for systematic error among the methods used. In the absence of systematic error, a 95% prediction interval predicts where the true concentrations of 95% of the samples of this SRM lie. The certified values were corroborated by analyses from nine Polish laboratories cooperating on the certification under the direction of T. Plebanski and J. Lipinski, Polish Committee for Standardization Measures, and Quality Control. The Polish laboratory work was supported by the Maria Sklodowska-Curie Joint Fund.

Table 1. Certified Values

<u>Element</u>	<u>wt. %</u>	<u>Element</u>	<u>μg/g</u>
Aluminum	7.50 ± 0.06	Antimony	7.9 ± 0.6
Calcium	1.89 ± 0.05	Arsenic	17.7 ± 0.8
Iron	3.50 ± 0.11	Barium	968 ± 40
Magnesium	1.51 ± 0.05	Cadmium	0.38 ± 0.01
Phosphorus	0.062 ± 0.005	Chromium	130 ± 4
Potassium	2.03 ± 0.06	Cobalt	13.4 ± 0.7
Silicon	29.66 ± 0.23	Copper	34.6 ± 0.7
Sodium	1.16 ± 0.03	Lead	18.9 ± 0.5
Sulfur	0.089 ± 0.002	Manganese	538 ± 17
Titanium	0.342 ± 0.024	Mercury	1.40 ± 0.08
		Nickel	88 ± 5
		Selenium	1.57 ± 0.08
		Silver	0.41 ± 0.03
		Strontium	231 ± 2
		Thallium	0.74 ± 0.05
		Vanadium	112 ± 5
		Zinc	106 ± 3

**Noncertified Values:** Noncertified values, shown in parentheses, are provided for information only. An element concentration value may not be certified if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available. Certified values for some of these elements will eventually be provided in a revised certificate when more data is available.

Table 2. Noncertified Values

<u>Element</u>	<u>wt. %</u>	<u>Element</u>	<u>μg/g</u>
Carbon	(1.2)	Cerium	(42)
		Cesium	(5.3)
		Dysprosium	(3.5)
		Europium	(0.9)
		Gallium	(14)
		Gold	(0.3)
		Hafnium	(3.7)
		Holmium	(0.54)
		Iodine	(5)
		Lanthanum	(23)
		Molybdenum	(2.0)
		Neodymium	(19)
		Rubidium	(96)
		Samarium	(3.8)
		Scandium	(12)
		Thorium	(11)
		Tungsten	(2)
		Uranium	(3)
		Ytterbium	(1.6)
		Yttrium	(18)
		Zirconium	(160)



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material 2710

#### Montana Soil

#### Highly Elevated Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. SRM 2710 is a highly contaminated soil that was oven-dried, sieved, and blended to achieve a high degree of homogeneity. A unit of SRM 2710 consists of 50 g of the dried material.

The certified elements for SRM 2710 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should not be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

#### NOTICE AND WARNINGS TO USERS

**Expiration of Certification:** This certification is valid for 5 years from the date of shipment from NIST. Should any of the certified values change before the expiration of the certification, purchasers will be notified by NIST. Return of the attached registration card will facilitate notification.

**Stability:** This material is considered to be stable; however, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser.

**Use:** A minimum sample weight of 250 mg (dry weight - see Instructions for Drying) should be used for analytical determinations to be related to the certified values on this Certificate of Analysis.

To obtain the certified values, sample preparation procedures should be designed to effect complete dissolution. If volatile elements (i.e., Hg, As, Se) are to be determined, precautions should be taken in the dissolution of SRM 2710 to avoid volatilization losses.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The overall direction and coordination of the analyses were under the chairmanship of M.S. Epstein and R.L. Waters, Jr., of the NIST Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills and J.S. Kane.

Gaithersburg, MD 20899  
August 23, 1993  
(Revision of certificate dated 10-30-92)

Thomas E. Gills, Acting Chief  
Standard Reference Materials Program

(over)



**Instructions for Drying:** When nonvolatile elements are to be determined, samples should be dried for 2 h at 110 °C. Volatile elements (i.e., Hg, As, Se) should be determined on samples as received; separate samples should be dried as previously described to obtain a correction factor for moisture. Correction for moisture is to be made to the data for volatile elements before comparing to the certified values. This procedure ensures that these elements are not lost during drying. The weight loss on drying has been found to be in the range of 1.7 to 2.3%.

**Source and Preparation of Material:** The U.S. Geological Survey (USGS), under contract to the NIST, collected and processed the material for SRM 2710. The soil was collected from the top 10 cm (4 in) of pasture land located at Longitude 112° 47' and Latitude 46° 01' along Silver Bow Creek in the Butte, Montana area. The site is approximately nine miles east of the local Anaconda plant and 6.5 miles south of settling ponds that feed the creek. The creek periodically floods, depositing sediment with high concentrations of copper, manganese, and zinc at the collection site. The material was shoveled from a 6.1 m x 6.1 m (20 ft x 20 ft) area into polyethylene bags in cardboard cartons for shipment to the USGS laboratory for processing.

The material was spread on 30.5 cm x 61 cm (1 ft x 2 ft) polyethylene-lined drying trays in an air drying oven and dried for three days at room temperature. The material was then passed over a vibrating 2 mm screen to remove plant material, rocks, and large chunks of aggregated soil. Material remaining on the screen was deaggregated and rescreened. The combined material passing the screen was ground in a ball mill to pass a 74 µm screen and blended for 24 h. Twenty grab samples were taken and measured for the major oxides using x-ray fluorescence spectrometry and for several trace elements using inductively coupled plasma atomic emission analysis to provide preliminary assessment of the homogeneity of the material prior to bottling. The material was bottled into 50 g units and randomly selected bottles were taken for the final homogeneity testing.

**Analysis:** The homogeneity, using selected elements in the bottled material as indicators, was assessed using x-ray fluorescence spectrometry and neutron activation analysis. In a few cases, statistically significant differences were observed, and the variance due to material inhomogeneity is included in the overall uncertainties of the certified values. The estimated relative standard deviation for material inhomogeneity is less than 2% for those elements for which homogeneity was assessed.

**Certified Values and Uncertainties:** The certified values are weighted means of results from two or more independent analytical methods, or the mean of results from a single definitive method, except for mercury. Mercury certification is based on cold vapor atomic absorption spectrometry used by two different laboratories employing different methods of sample preparation prior to measurement. The weights for the weighted means were computed according to the iterative procedure of Paule and Mandel (NBS Journal of Research 87, 1982, pp. 377-385). The stated uncertainty includes allowances for measurement imprecision, material variability, and differences among analytical methods. Each uncertainty is the sum of the half-width of a 95% prediction interval and includes an allowance for systematic error among the methods used. In the absence of systematic error, a 95% prediction interval predicts where the true concentrations of 95% of the samples of this SRM lie. The certified values were corroborated by analyses from nine Polish laboratories cooperating on the certification under the direction of T. Plebanski and J. Lipinski, Polish Committee for Standardization, Measures, and Quality Control. The Polish laboratory work was supported by the Maria Sklodowska-Curie Joint Fund.

Table 1. Certified Values

<u>Element</u>	<u>wt. %</u>		<u>Element</u>	<u>μg/g</u>	
Aluminum	6.44	± 0.08	Antimony	38.4	± 3.0
- Calcium	1.25	± 0.03	Arsenic	626	± 38
Iron	3.38	± 0.10	Barium	707	± 51
Magnesium	0.853	± 0.042	Cadmium	21.8	± 0.2
Manganese	1.01	± 0.04	Copper	2950	± 130
Phosphorus	0.106	± 0.015	Lead	5532	± 80
Potassium	2.11	± 0.11	Mercury	32.6	± 1.8
Silicon	28.97	± 0.18	Nickel	14.3	± 1.0
Sodium	1.14	± 0.06	Silver	35.3	± 1.5
Sulfur	0.240	± 0.006	Vanadium	76.6	± 2.3
Titanium	0.283	± 0.010	Zinc	6952	± 91

Noncertified Values: Noncertified values, shown in parentheses, are provided for information only. An element concentration value may not be certified if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available. Certified values for some of these elements will eventually be provided in a revised certificate when more data is available.

Table 2. Noncertified Values

<u>Element</u>	<u>wt. %</u>	<u>Element</u>	<u>μg/g</u>
Carbon	(3)	Bromine	(6)
		Cerium	(57)
		Cesium	(107)
		Chromium	(39)
		Cobalt	(10)
		Dysprosium	(5.4)
		Europium	(1)
		Gallium	(34)
		Gold	(0.6)
		Hafnium	(3.2)
		Holmium	(0.6)
		Indium	(5.1)
		Lanthanum	(34)
		Molybdenum	(19)
		Neodymium	(23)
		Rubidium	(120)
		Samarium	(7.8)
		Scandium	(8.7)
		Strontium	(240)
		Thallium	(1.3)
		Thorium	(13)
		Tungsten	(93)
		Uranium	(25)
		Ytterbium	(1.3)
		Yttrium	(23)



REC'D 4-15-77

# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material 2711

#### Montana Soil

#### Moderately Elevated Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. SRM 2711 is a moderately contaminated soil that was oven-dried, sieved, and blended to achieve a high degree of homogeneity. A unit of SRM 2711 consists of 50 g of the dried material.

The certified elements for SRM 2711 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should not be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100-mg samples.

#### NOTICE AND WARNINGS TO USERS

**Expiration of Certification:** This certification is valid for 5 years from the date of shipment from NIST. Should any of the certified values change before the expiration of the certification, purchasers will be notified by NIST. Return of the attached registration card will facilitate notification.

**Stability:** This material is considered to be stable, however, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser.

**Use:** A minimum sample weight of 250 mg (dry weight - see Instructions for Drying) should be used for analytical determinations to be related to the certified values on this Certificate of Analysis.

To obtain the certified values, sample preparation procedures should be designed to effect complete dissolution. If volatile elements (i.e., Hg, As, Se) are to be determined, precautions should be taken in the dissolution of SRM 2711 to avoid volatilization losses.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The overall direction and coordination of the analyses were under the chairmanship of M.S. Epstein and R.L. Watters, Jr., of the NIST Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by T.E. Gills and J.S. Kane.

Gaithersburg, MD 20899  
October 30, 1992

William P. Reed, Chief  
Standard Reference Materials Program

(over)

Instructions for Drying: When nonvolatile elements are to be determined, samples should be dried for 2 h at 110 °C. Volatile elements (i.e., Hg, As, Se) should be determined on samples as received; separate samples should be dried as previously described to obtain a correction factor for moisture. Correction for moisture is to be made to the data for volatile elements before comparing to the certified values. This procedure ensures that these elements are not lost during drying. The approximate weight loss on drying has been found to be in the range of 1.5 to 2.2%.

Source and Preparation of Material: The U.S. Geological Survey (USGS), under contract to the NIST, collected and processed the material for SRM 2711. The material is an agricultural soil collected in the till layer (upper 15.2 cm (6 in)) of a wheat field. The soil from a 3.05 m x 3.05 m (10 ft x 10 ft) area was shoveled into 0.114 m<sup>3</sup> (3 gal) plastic pails for shipment to the USGS laboratory for processing.

The material was spread on 30.5 cm x 61 cm (1 ft x 2 ft) polyethylene-lined drying trays in an air drying oven and dried for three days at room temperature. The material was then passed over a vibrating 2-mm screen to remove plant material, rocks, and large chunks of aggregated soil. Material remaining on the screen was deaggregated and rescreened. The combined material passing the screen was ground in a ball mill to pass a 74- $\mu$ m screen and blended for 24 h. Twenty grab samples were taken and measured for the major oxides using x-ray fluorescence spectrometry and for several trace elements by using inductively coupled plasma atomic emission analysis to provide preliminary assessment of the homogeneity prior to bottling. The material was bottled into 50-g units and randomly selected bottles were taken for the final homogeneity testing.

Analysis: The homogeneity, using selected elements in the bottled material as indicators, was assessed using x-ray fluorescence spectrometry and neutron activation analysis. In a few cases, statistically significant differences were observed, and the variance due to material inhomogeneity is included in the overall uncertainty of the certified value. The estimated relative standard deviation is less than 3 % for those elements for which homogeneity was assessed.

Certified Values and Uncertainties: The certified values are weighted means of results from two or more analytical methods, or the mean of results from a single definitive method, except for mercury. Mercury certification is based on cold vapor atomic absorption spectrometry used by two different laboratories employing different methods of sample preparation prior to measurement. The weights for the weighted means were computed according to the iterative procedures of Paule and Mandel (NBS Journal of Research 87, 1982, pp. 377-385). The stated uncertainty includes allowances for measurement imprecision, material variability, and differences among analytical methods. Each uncertainty is the sum of the half-width of a 95 % prediction interval and includes an allowance for systematic error among the methods used. In the absence of systematic error, a 95 % prediction interval predicts where the true concentrations of 95 % of the samples of this SRM lie.

Table 1. Certified Values

<u>Element</u>	<u>wt. %</u>		<u>Element</u>	<u>μg/g</u>	
Aluminum	6.53	± 0.09	Antimony	19.4	± 1.8
Calcium	2.88	± 0.08	Arsenic	105	± 8
Iron	2.89	± 0.06	Barium	726	± 38
Magnesium	1.05	± 0.03	Cadmium	41.70	± 0.25
Phosphorus	0.086	± 0.007	Copper	114	± 2
Potassium	2.45	± 0.08	Lead	1162	± 31
Silicon	30.44	± 0.19	Manganese	638	± 28
Sodium	1.14	± 0.03	Mercury	6.25	± 0.19
Sulfur	0.042	± 0.001	Nickel	20.6	± 1.1
Titanium	0.306	± 0.023	Selenium	1.52	± 0.14
			Silver	4.63	± 0.39
			Strontium	245.3	± 0.7
			Thallium	2.47	± 0.15
			Vanadium	81.6	± 2.9
			Zinc	350.4	± 4.8

**Noncertified Values:** Noncertified values, shown in parentheses, are provided for information only. An element concentration value may not be certified, if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available. Certified values for some of these elements will eventually be provided in a revised certificate when more data is available.

Table 2. Noncertified Values

<u>Element</u>	<u>wt. %</u>	<u>Element</u>	<u>μg/g</u>
Carbon	(2)	Bromine	(5)
		Cerium	(69)
		Cesium	(6.1)
		Chromium	(47)
		Cobalt	(10)
		Dysprosium	(5.6)
		Europium	(1.1)
		Gallium	(15)
		Gold	(.03)
		Hafnium	(7.3)
		Holmium	(1)
		Indium	(1.1)
		Iodine	(3)
		Lanthanum	(40)
		Molybdenum	(1.6)
		Neodymium	(31)
		Rubidium	(110)
		Samarium	(5.9)
		Scandium	(9)
		Thorium	(14)
		Tungsten	(3)
		Uranium	(2.6)
		Ytterbium	(2.7)
		Yttrium	(25)
		Zirconium	(230)

## EXPANDED DELIVERABLE DATA REVIEW SHEET

LOT # SRM1 - Analyzed by TJA61 ICP

Fraction: VOA BNA PEST/PCB METALS WET CHEM (CIRCLE ONE)

Date Reviewed: 5-17-96Reviewed By: Susan Palmer

QA/QC Comments (Please note if no problems encountered):

Certain elements were not reported due to the instrument being out of control or due to studies being incomplete at the time of analysis.

Certain TRACE level elements were not reported due to the instruments inability to reach IDC's at the Level of the SEM standard.

☐

Form VI represents the duplication of the actual MS/MSD results.

☐

No Form VIII included - No MSA's performed.

☐

No Form IX included - No serial dilution performed.

☐

No Form V or Form VI provided - No MS/MSD performed.

☐

No Form IX provided for this lot. The serial dilution was performed on a sample in lot \_\_\_\_\_ from this client and from the same QC batch as the samples in this lot.

Level IIC Review:

Calculations: SPUnits:   Prep/Anal. Date:   Level I done:   Level IIa done: SP

5-17-96

Susan Palmer

7  
LABORATORY CONTROL SAMPLE

Client:

SDG No.: SRM1

Aqueous LCS Source: \_\_\_\_\_

FORM VII - IN

LABORATORY CONTROL SAMPLE

Client:

SDG No.: SRM1

Aqueous LCS Source: \_\_\_\_\_

FORM VII - IN





7  
LABORATORY CONTROL SAMPLE

Client:

SDG No. : SRM1

Aqueous LCS Source: \_\_\_\_\_

[illegible]

7  
LABORATORY CONTROL SAMPLE

Client: \_\_\_\_\_

SDG No.: SRM1

Aqueous LCS Source: \_\_\_\_\_

[illegible]

7  
LABORATORY CONTROL SAMPLE

Client:

SDG No.: SRM1

Aqueous LCS Source: \_\_\_\_\_

FORM VII - IN

7  
LABORATORY CONTROL SAMPLE

Client:

SDG No.: SRM1

Aqueous LCS Source: \_\_\_\_\_

[illegible]

7  
LABORATORY CONTROL SAMPLE

Client:

SDG No. : SRM1

Aqueous LCS Source: \_\_\_\_\_

[illegible]

7  
LABORATORY CONTROL SAMPLE

Client: \_\_\_\_\_

SDG No.: SRM1

Aqueous LCS Source: \_\_\_\_\_

[illegible]



Setting the Standards for Innovative  
Environmental Solutions

May 21, 1996

Mr. Bruce Roberts  
BASF Corporation  
1609 Biddle Ave.  
Wyandotte, MI 48192

Dear Mr. Roberts:

The following are Environmental Standards, Inc.'s responses to comments prepared by Mr. David A. Payne of U.S. EPA, Region V, regarding Environmental Standards' Standard Operating Procedures (SOPs) for data review/validation of analytical data generated as part of the BASF investigation of the Northworks Facility.

Environmental Standards' responses to the Region V comments will follow the format of restating the Region V comment followed by Environmental Standards' response in *italic* typeset. The issues and responses are as follows:

1. "The ESI SOPs for metals and cyanide should not be significantly effected by extra Appendix IX elements. Appendix IX includes the element tin, but the CLP Target Analyte List (TAL) does not. Tin has poor analytical performance compared to other ICP elements, but I have never known tin to ever be a factor at any RCRA or Superfund site in Region 5. The ESI SOPs may not specifically address the ICP Trace instrument, but ESI staff are known to be knowledgeable on its use. When Quanterra completes its work on cyanide, the laboratory should meet, or exceed the criteria established by ESI for acceptable cyanide data."

*The addition of tin to the list of analytes and the use of Trace ICP analysis does not warrant any special notation or addition to the validation SOP and Environmental Standards agrees that these issues will not affect the use of the SOPs in the evaluation of the analytical data.*



2. "The ESI SOPs were written in mid-1995, for use of less than 30% for the %RSD of initial calibration for Methods 8240, 8260, and 8270. SW-846, Update II, now requires less than 15% for initial calibration. I briefly looked at the Method 8260A Quanterra SOP. This specifies 80% of the volatile target compounds shall have an initial calibration with a % RSD less than 15%. This is a unique twist."

*During the review of the analytical data, Environmental Standards will follow the data review/validation SOPs that were submitted to Region V. However, methodology updates, such as the one pointed out here, will be incorporated into the data review/validation SOP used by Environmental Standards.*

3. "ESI's SOP for volatiles is written for a 5 mL purge volume, not the 25 mL purge volume of Method 8260. Water miscible volatiles (acetone, acrolein, etc.) behave differently as discussed in lab evaluation memo for Quanterra, for 5 mL and 25 mL sample aliquots."

*Environmental Standards agrees that some compounds behave differently with 25 mL purge volumes versus 5 mL purge volumes. However, the evaluation approach that is defined within the SOP provides details of how data quality will be accessed. This approach is not effected by the difference in purge volume. For example, water miscible compounds like acrolein will purge less effectively in 25 mL than in 5 mL. However, for the interpretation of data quality as a function of evaluating the relative response factors (RRFs), the "rules" as defined in the SOP are appropriately the same; any compounds with an RRF of  $<0.05$  will result in "not detected" results being flagged "R" and any positive results being flagged "J".*

4. "ESI's SOPs use a minimum relative response factor (RRF) of 0.05 for volatiles, semi-volatiles and SPCC criteria. Quanterra uses less than 0.3 for volatiles SPCC criteria. My observations have been:"
  - a. "Poor performance for water miscible volatiles generally has occurred when the target volatiles RRF has been less than 0.05. Certain volatiles (acrolein, acetonitrile, etc.) will never have a RRF greater than 0.05. Acrylonitrile and acetone exhibit borderline behavior. The 0.05 criteria is reasonable."



*It appears that there is agreement that 0.05 is an acceptable RRF criteria for use in validation. Environmental Standards agrees that compounds like acrolein and acrylonitrile typically do not exhibit "healthy" RRFs by this method. Quite often, qualification or rejection of certain data has nothing to do with laboratory performance and much to do with method performance. It is not clear if what is being requested is to be more "forgiving" for method performance problems*

- b. "The first internal standard used for volatiles (bromochloromethane or pentafluoromethane) can have low response causing a larger RRF than calculated for other internal standards."

*This is a true observation as the individual target analyte response factors are calculated relative to the associated internal standard. It does not appear that a modification or addition to the SOP is being requested.*

- c. "The Quanterra lab evaluation report identified organic compounds that will not, or may not, meet the 0.05 RRF. Four or five Appendix IX organic compounds may not be detected at all."

*Please see Response 4a.*

5. "The ESI SOP for sulfide considers sample distillation for sulfide. Quanterra will be separating sulfide by precipitation with zinc hydroxide. ESI and Quanterra analytical methods are different."

*Environmental Standards will revise the data review/validation SOP associated with sulfide analysis to reflect the specific analytical procedure performed by Quanterra.*

6. "The ESI SOPs discuss calibration factors for initial calibrations in Methods 8080 and 8150. Quanterra uses a second order regression for initial calibration of single component compounds in Method 8080 and 8150. Quanterra uses calibration factors for Aroclors, tech. chlordane, and toxaphene in Method 8080. ESI is familiar with second order regression behavior."



*Environmental Standards personnel are familiar with second order regression analysis and will appropriately evaluate the initial calibration of single component compounds in Methods 8080 and 8150.*

With the exception of updating the sulfide data validation SOP to include additional items unique to the specific analytical method being used by Quanterra, it does not appear that any further action is being required of Environmental Standards. If you have any questions/comments, please do not hesitate to call.

Sincerely;



George R. Mussoline  
Senior Quality Assurance Chemist/  
Project Manager



Rock J. Vitale, CPC  
Director of Chemistry

GRM/RJV/hb



May 17, 1996

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (DRE-8J)  
77 West Jackson Street  
Chicago, Illinois 60604

RE: Additional response to EPA's conditional approval letter dated March 11, 1996

Dear Ms. Sharrow:

Over the past couple of months, QES personnel have been working closely with Mr. David Payne (USEPA) to understand and to resolve the SOP issues. The SOPs that Mr. Payne evaluated were mostly facility SOPs and reflected Update I methods. Since that time, QES has begun using Update II methods and is using more corporate SOPs. QES will add addenda to their corporate SOPs, as required, to satisfy Region V requirements. Also, QES has renamed many of their facility SOPs.

In some cases, the SOP specified in the tables in Section 7 may not be the one specified in your conditional approval letter. A good example of this is the ICP analysis; your letter specifies the use of SOP NC-MT-0006. QES stopped using this SOP last November; now they use SOP CORP -MT-0001 which is a corporate SOP and reflects Update II. QES is a commercial lab and can not have separate SOPs for every client. The SOPs specified in Section 7 are the SOPs that are actually in use. We believe that the quality of the data could suffer and the chances for error are much greater if EPA requires QES to alter their normal routine and use the older, outdated Update I methods. One possible exception for altering the normal routine is for Prussian Blue cyanide analysis because of its unique matrix. BASF requests that QES be allowed to use the SOPs that are in daily use at the lab. If you require the use of Update I methods, please supply specific reasons why the older SOP is better.

Below is our additional response to EPA's conditional approval dated March 11, 1996. With the exception of cyanide, all issues in the "Lab Audit Conditions for QAPP Approval Summary" document have been addressed. In addition, many of the recommendations in the lab audit evaluation have been acted upon.

Corrective Measures for Method 8270

Aramite and p-phenylenediamine have been removed from the QAPP. Hexachlorophene has been changed to Method 8150B. Dinoseb has been removed from Method 8150B and will be analyzed using Method 8270B.

Conditional Approval Items

**B.1.a - update tables in facility SOPs for method 8240 - soil and 8260 - water**

EPA reviewed and commented on Quanterra (QES) SOPs LM-WALN-3020 and NC-MS-0002. QES has replaced these two facility SOPs with corporate SOP CORP-MS-0002 and has revised/added tables as addenda. Addenda Tables 1 and 3 give revised reporting limits for 25 ml aliquots and replace only one column of Table 1 and 3 in the corporate SOP. Addenda Tables 2 and 4 present primary standard calibration levels for a 25 ml purge and replace the corporate SOP Tables 2 and 4. Addenda Tables 18 and 19 are new tables which state the primary and Appendix IX retention times on the two columns being used. The characteristic ions are presented in corporate SOP Table 13.

**B.1.b - experimental reference spectra for data validation**

An addendum has been added to the corporate SOP stating that it may be appropriate to supply laboratory generated mass spectra instead of NIST mass spectra for data validation purposes.

**B.1.c - re-evaluate standard concentrations for water miscible volatiles**

As stated in B.1.a above, the calibration concentrations for the 25 ml aliquots have been revised. Examples of acetone and 2-butanone spectra from recent 25 ml calibration standards from various instruments are attached for your review. These spectra should demonstrate that adequate sensitivity is routinely being achieved in a 25 ml purge to meet the qualitative requirements of the method for these water miscible compounds, even at concentrations well below the reporting limits that are required for this project.

**B.1.d - establish conditions to obtain appropriate experimental mass spectra for acrolein, acetonitrile, propionitrile, and dibromochloropropane**

QES has separated allyl chloride and acetonitrile into different calibration standards. QES has also revised the standard calibration levels for acrolein, acetonitrile, propionitrile, and dibromochloropropane which are shown in addenda Tables 2 and 4. In addition, the concentration of ethyl acetate in the calibration standards has been reduced to lessen any potential for interference with propionitrile.

**B.4.b - alternate method for hexachlorophene**

Hexachlorophene will be analyzed by Method 8150B. This change is reflected in TABLE 7-4 and method detection limits and targeted quantitation limits will be included in the table when they are determined.

**B.5 - report cis 1,2-dichloroethene, a-chlordane, and g-chlordane with Appendix IX results**

Cis 1,2-dichloroethene, a-chlordane, and g-chlordane will be reported with Appendix IX results. TABLE 7-4 has been revised to include these compounds.

**B.6 - report aramite and p-phenylene diamine as not detectable and delete OPPs from QAPP**

Aramite, p-phenylene diamine, and OPPs have been removed from the QAPP tables.

**C.1 - delete sulfide in soil from the RFI**

The sulfide in soil parameter has been deleted from the QAPP.

**C.2 - additional information for cyanide - Prussian Blue area**

Additional information has been received from Mr. David Payne, but no decision has been made. Further discussions with Mr. Payne are required. Attention has been focused in resolving the cyanide issues for analyses for areas outside of the Prussian Blue area, but will now be shifted to the Prussian Blue area.

**C.3 - utilize facility SOP for Method 8150**

QES will utilize the facility SOP LM-WALN-4110 for the extraction and will use the corporate SOP CORP-GC-0001 for the analysis.

**C.4 - delete SOP CORP-MT-0003 for GFAA**

SOP CORP-MT-0003 for GFAA has been deleted from the QAPP. GFAA will not be used for any analyses during this RFI.

**Additional Items**

In your letter dated April 18, you requested that the third sentence in item number 3, Section 4 page 7 of the QAPP be deleted. Even though EPA had approved that sentence previously, it has been deleted.

**B.2.a - ICP Method 6010A and ICP Trace Method 6010A to be used for metals**

QES will not be using GFAA for this project and references to GFAA have been eliminated from the QAPP. At a minimum the ICP Trace will be used for As, Pb, Tl, Se, and Sb in water and As, Se, Tl, and Sb in soils. QAPP tables have been revised.

**B.2.b - soils for mercury, cyanide, and metals to be dried/homogenized**

Soils for mercury, cyanide, and metals analyses will be dried and homogenized prior to analysis. QES has developed SOP NC-IP-0001 for this procedure and is shown in TABLE 7-1.

**B.2.c - rewrite QAPP tables to reflect use of SOP NC-MT-0006**

Facility SOP NC-MT-0006 has been replaced with corporate SOP CORP-MT-0001. Since SOP NC-MT-0006 is no longer used, it has been deleted from the QAPP.

**B.2.d - use of NIST SRM soils numbers 2709, 2710, and 2711**

QES purchased the above listed soils and they arrived at the lab on April 15. QES will submit the results from the study to EPA during the week of May 20.

**B.3.a - establish acceptance criteria for surrogate spike recoveries for Method 8150**

Quality control is shown in Section 9 of the corporate SOP CORP-GC-0001 and in Section 11 of the facility SOP LM-WALN-4110. The acceptance criteria for surrogate spike recoveries are not in the SOPs but are shown in QAPP TABLE 3-2.

**B.3.b - decrease concentrations used for surrogate and matrix spikes**

The facility SOP (LM-WALN-4110) rather than the corporate SOP will be used for herbicide extractions and the facility SOP contains the correct concentrations; the spiking is done during the preparation. Water samples will be analyzed straight and soil samples will be analyzed at a 1:10 dilution.

**B.4.a - alternative method for 1,4-dioxane**

1,4-dioxane will be analyzed by Method 8270B; this change is reflected in TABLE 7-4.

TABLE 7-4 page 15 still lists "ND" for the method detection limits in Methods 8150B and 8015A; QES is in the process of determining the limits.

The signature sheet has been signed by QES and is in the process of being signed by ESE. As soon as it signed, it will be sent to you for EPA signature.

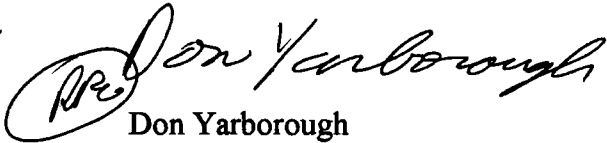
ESI is currently reviewing Mr. Payne's comments concerning the data validation that were attached to your April 23 letter. A response will be submitted to you soon.

A revised schedule will be sent under separate cover during the week of May 20.

Attached are a page change summary and a lab SOP change summary.

In addition, a status summary of the recommendations in Mr. Payne's laboratory evaluation is attached. This is being included for reference only. Receiving full EPA approval is based upon correcting conditions specified in your document "Lab Audit Conditions for QAPP Approval Summary" except where the summary references the laboratory evaluation.

Sincerely,



Don Yarborough  
Wyandotte Site Manager

Attachments

z:\winword\05176.ltr

cc: B. Roberts - BASF  
R. Veenstra - ESE  
R. Vitale - ESI  
R. Blayer - MDEQ Lansing  
L. Aubuchon - MDEQ Livonia  
J. Russell - MDEQ Livonia  
T. Himes - Quanterra



### Page Change Summary

QAPP title sheet -- page revised to reflect latest revision, ESE project number, and current date.

QAPP signature page -- page was revised to reflect latest revision and current date.

QAPP Section 1 page 48 -- Item 8 has been revised to remove reference to aramite and to add a reference to lab SOP CORP-MS-0002. Details of a TIC search are specified in the lab SOP.

QAPP TABLE 1-3 -- Note 2 was added.

QAPP TABLE 3-1 -- Method numbers were revised to reflect Update II, the recovery limits for 2,4-D were corrected, ICP metals listing was revised to reflect ICP Trace, furnace metals were removed, and recovery limits for cyanide and sulfide were revised.

QAPP TABLE 3-2 -- Method numbers were revised to reflect Update II and a typographical error was corrected for phenol.

QAPP Section 4 page 7 -- Third sentence in item number 3 was deleted.

QAPP TABLE 6-1 -- Method numbers were revised to reflect Update II, Method 7000 GFAA was deleted, acceptance criteria were revised, the relative position of some items in the table were shifted because of the deletion of Method 7000.

QAPP Section 7 -- Because reference to GFAA was deleted, the section was reduced from 17 to 16 pages.

QAPP TABLE 7-1 -- Arsenic and selenium Method 7060 was deleted, metals GFAA Method 3020A was deleted, inorganics grinding and chlorinated herbicides were added, Method numbers were revised to reflect Update II, lab SOP LM-WALN-2530 was replaced with NC-IP-0004, some lab SOPs were deleted, note numbers were revised, note 4 was added, note 5 was revised, and reference to GFAA was deleted.

QAPP TABLE 7-2 page 5 -- References to GFAA methods were deleted, facility SOP NC-MT-0006 was deleted, facility SOPs for mercury were replaced with corporate SOPs, SOP LM-WALN-1330 was replaced with NC-WC-0060, and Method 9045B was revised to 9045C.

QAPP TABLE 7-2 page 6 -- Method numbers were revised to reflect Update II, SOP LM-WALN-4110 was moved to TABLE 7-1, facility SOP LM-WALN-4060 was deleted, SOP LM-WALN-1141 was replaced with NC-WC-0031, and note 2 was revised.

QAPP TABLE 7-4 -- Method numbers were revised to reflect Update II, method detection limits were revised, cis-1,2-dichloroethene was added on page 10, OPPs were deleted, aramite was deleted, 1,4-dioxane was added on page 12, hexachlorophene was added on page 15, nonhalogenated volatiles other than PG and PO were deleted from Method 8015A, silver was moved from ICP Trace to ICP, and detection limits for cyanide and sulfide were revised.

QAPP Section 8.2 page 3 -- Reference to GFAA was deleted and items were re-numbered.

QAPP Section 9.2.3 page 3 inorganic analyses -- Reference to GFAA was deleted and items were re-numbered.

QAPP TABLE 11-1 -- Reference to GFAA was deleted and the relative position of items were shifted and ICP Trace was added on page 5.

### Lab SOP Change Summary

Remove Appendix C title sheet and table of contents and replace with revised copy.

SOP	STATUS
NC-SC-0005	Replace with later revision
NC-QA-0002	No change
NC-SC-0001	No change
NC-WC-0004	No change
NC-IP-0002	Remove - no longer required for RFI
NC-IP-0001	Add to QAPP
NC-IP-0003	Replace with later revision
LM-WALN-4110	No change
LM-WALN-2500	Remove - no longer required for RFI
LM-WALN-2530	Remove and replace with NC-IP-0004 and place behind NC-IP-0003
NC-WC-0032	Replace with later revision and relocate to behind NC-IP-0004
NC-OP-0009	Remove - no longer required for RFI
NC-OP-0013	Remove - no longer required for RFI
CORP-OP-0001	Replace with later revision
LM-WALN-5020	Remove - no longer required for RFI
LM-WALN-5060	Remove - no longer required for RFI
NC-IP-0005	No change
CORP-MT-0001	No change
NC-MT-0006	Remove - no longer required for RFI

CORP-MT-0003	Remove - no longer required for RFI
NC-MT-0002	Remove - no longer required for RFI
NC-MT-0005	Remove and replace with CORP-MT-0007
NC-MT-0004	Remove and replace with CORP-MT-0005 and relocate in front of CORP-MT-0007
LM-WALN-1330	Remove and replace with NC-WC-0060
NC-WC-0010	Replace with later revision
LM-WALN-3020	Remove - no longer required for RFI
NC-MS-0002	Remove and replace with CORP-MS-0002 including addenda
NC-MS-0004	Remove and replace with CORP-MS-0001 including addendum
CORP-GC-0001	Replace with later revision and relocate to behind CORP-MS-0001
LM-WALN-4060	Remove - no longer required for RFI
LM-WALN-1141	Remove and replace with NC-WC-0031
NC-GC-0021	Replace with later revision
NC-GC-0022	Replace with later revision

#### D. Payne Evaluation of Laboratory Services

- II.A. - OPPs have been deleted from QAPP.
- II.B.R. - 1,4-dioxane added to Method 8270 and shown in TABLE 7-4.
- II.C.R1. - Aramite and p-phenylenediamine deleted from Method 8270.
- II.C.R2. - Hexachlorophene added to Method 8150; method detection limits and targeted quantitation limits are being determined by QES.
- II.C.R1. - Pentachlorophenol kept as part of Methods 8150 and 8270.
- II.C.R2. - Dinoseb has been removed from Method 8150 and added to Method 8270.
- II.D.1. - cis 1,2-dichloroethene added to Method 8240 (already part of Method 8260) and shown in TABLE 7-4.
- II.D.2. - a-chlordane and g-chlordane have been added to TABLE 7-4.
- III.A.4. - Only the revised corporate SOP CORP-GC-0001 will be used. No specific criteria was given for using the facility SOP other than the corporate SOP is more generic.
- III.B.R1 - Representative soil samples will be dried and homogenized. This procedure is specified in facility SOP NC-IP-0001 and is listed in TABLE 7-1. The facility Sop will be used in conjunction with the corporate SOP.
- III.B.R2. - ICP Trace will be used for As, Pb, Se, Tl, and Sb in water and As, Se, Tl, and Sb in soils. Copper and silver will not be analyzed using ICP Trace.
- III.B.R3. - QAPP tables have been rewritten to reflect recommendations 1 and 2.
- III.B.R4. - The interference check sample solution is purchased from a vendor. It has been verified that contamination has been introduced from the vendor; however, the contamination does not impact the data in any way.
- III.B.R5. - QES has purchased NIST soils SRMs 2709, 2710, and 2711 and is in the process of testing them.
- III.B.R6. - Antimony will be analyzed by ICP Trace.
- III.B.R7. - Discussions between QES and USEPA to determine the appropriate methodologies for the Prussian Blue area have begun and will continue when Mr. Payne returns from vacation.

III.C.1.a.R.1 - The tables in the SOPs will not be rewritten so that each SOP is specific to Appendix IX compounds. QES developed SOPs to be used for all clients and they will not be changing them to be specifically written for this RFI. Tables 18 and 19 have been added to list retention times for the two columns being used. The characteristic ions are already shown in Table 13 of corporate SOP CORP-MS-0002.

No → III.C.1.a.R2. - Since the RFI is only focusing on Appendix IX compounds, data validation should not be concerned with non-Appendix IX compounds.

No → III.C.1.b.R. - As stated earlier, an addendum has been added to the corporate SOP stating that it may be appropriate to supply laboratory generated mass spectra instead of NIST mass spectra for data validation purposes.

? → III.C.1.c.R. - QES has added addendum tables Tables 1 and 3 giving revised reporting limits for 25 ml aliquots.

III.C.1.d.R. - To be evaluated.

III.C.1.e.R1. - Acetonitrile and allyl chloride have been separated into separate calibration standards.

III.C.1.e.R2. - The calibration standard concentrations used for acrolein and propionitrile have been revised.

III.C.1.e.R3. - The concentration of ethyl acetate in the calibration standards has been reduced to lessen any potential for interference with propionitrile.

III.C.1.f.R1. - See item B.1.d. in the cover letter.

✓ III.C.1.f.R2. - The corporate SOP has been revised to use ions 157 or 155 when quantifying 1,2-dibromo-3-chloropropane.

III.C.1.h.R1. - Not a recommendation

✓ III.C.1.h.R2. - TIC search is specified in corporate SOP CORP-MS-0002

→ III.C.1.h.R3. - Not a recommendation

III.C.1.h.R4. - QES will consider this recommendation.

III.C.2.a.R1. - OPPs have been deleted from the QAPP tables.

III.C.2.a.R2. - The correct nomenclature for 5-nitro-o-toluidine will be used.

III.C.2.a.R3. - Proprietary compound No. 13 is no longer used in Method 8270.

III.C.2.a.R4. - QES has removed this constituent from the list of target analytes.

III.C.2.a.R5. - Hexachlorophene has been deleted from Method 8270 and added to Method 8150.

III.C.2.a.R6. - Aramite has been deleted.

III.C.2.a.R7. - The doublet peak for Bis(2-Chloroisopropyl) ether no longer exists in the calibration standards currently in use.

III.C.2.a.R8. - The sentences "Care must be taken when evaluating data for the presence of phenol. Aniline at high concentrations could provide a 'false positive' for phenol because the two compounds co-elute and have a common secondary ion." have been added as an addendum to SOP CORP-MS-0001 which is GC/MS based on method 8270B. In addition, the analysts have been trained to recognize these coelution issues.

III.C.2.a.R9. - Should either constituent be detected, the project narrative will notify the client that coelution exists.

III.C.2.a.R10. - OPPs have been deleted from the project.

III.C.2.a.R11. - These ions have been added to the mass spectral library for symmetrical trinitrobenzene.

III.C.2.a.R12. - To be evaluated.

III.C.3.R1. - Hexachlorophene has been deleted from Method 8270 and added to Method 8150.

III.C.3.R2. - See item B.3.b. in the cover letter.

III.C.3.R3. - See item B.3.a. in the cover letter.

III.C.3.R4. - Pentachlorophenol will be maintained as a target compound for Method 8150.

III.C.3.R5. - The typographical error for 2,4-D in TABLE 3-1 has been corrected.

III.D.1.R. - Sulfide in soil has been deleted from the QAPP.

III.D.2.R - The cyanide control limits have been revised and are being reviewed by EPA.

III.D.3R. - Use of GFAA is no longer required by the RFI and its use has been deleted from the QAPP.

III.D.4R. - The facility SOP will be utilized for organic preparation.



Thomas M. Himes  
Project Manager



Quanterra Incorporated  
4101 Shuffel Drive, NW  
North Canton, Ohio 44720

216 497-9396 Telephone  
216 497-0772 Fax

May 6, 1996

Dave Payne  
U.S. EPA  
77 West Jackson Blvd.  
DRT - 14J  
Chicago, IL 60604-3590

Re: BASF RFI Cyanide Analyses / Method Modifications for Prussian Blue Area Samples

Dear Dave:

In response to your recently submitted documents on alternative cyanide methods, this correspondence serves to offer Quanterra Environmental Services' recommendations for your consideration.

Upon review of the literature and from our various telephone conversations, Dr. Mark Bruce feels that the following issues must be considered:

Since there is the probability that both positive and negative interferences will be present in samples from the Prussian Blue area, it is unlikely that we will be able to produce interference free data using our current CN SOP. Unfortunately, some of the ways to deal with one interference may make others worse; however, we could implement a few interference options from the reference methods that we have not used in recent history.

The four main concerns about the solid samples are as follows:

1) Some iron cyanide complexes are very stable (or insoluble) under standard acidic distillation conditions. This interference would tend to produce a negative bias to the total CN results. These CN complexes can be dissolved under basic conditions (Standard Methods mentions this option). We can then acidify and distill, but the extra basic dissolution step will probably add a day to the analytical process. The basic dissolution step may also convert CN to SCN if polysulfide is present. Since many sulfur compounds are present at the site this conversion interference is likely for some samples. One of the research papers mentions a basic dissolution step that is not as aggressive as the Standard Methods option. Although it gives few details, it claims to produce fewer interferences.

Recent distillations of ferricyanide using our standard process produced recoveries of 110, 106 and 103%. So it would appear that this particular CN complex will not be a problem.

Dave Payne

May 6, 1996

Page 2

2) How well will a 1g subsample represent the original sample? These samples have been described as rubble, so obtaining a representative sample may be a valid concern. If we use a sample that has been dried and ground to a free flowing powder, we would get better precision on "duplicate analyses" but most HCN (if in the original sample) would probably be lost. According to some of the reference papers, some samples may have as much as 2% CN. This presents an obvious safety issue for all analysts who work with the samples. It also means that we may tax the absorbing capacity of the distillate receiver solution on the CN rack like never before. There should be enough NaOH in our 0.25M solution but it has never been tested at this CN concentration.

3) Thiocyanate may be converted to CN under some distillation conditions. Spiking 2 and 100 ppm SCN into DI water and doing our normal acidic distillation produced CN readings of 16 and 3 ppb respectively. Thus, the inherent interference in the midi distillation is small. Presumably the sulfamic acid retards the conversion of SCN to CN under acidic conditions. One of the papers claimed the effectiveness of sulfamic acid was not universal across all matrices studied. Thus, it is possible that some BASF samples might cause problems in this area. Various types of sample prep using complexing agents and mildly acidic conditions supposedly handled this interference better but none of these approaches have been validated sufficiently. Performing a SCN test in addition to the total CN might provide an idea as to whether or not the SCN to CN positive interference should be considered when interpreting the data for an individual sample.

4) Oxidized products of sulfide can convert CN to SCN under basic conditions (i.e. a preserved sample or the special dissolution suggested above in #1). Also, sulfide can distill over with the CN and interfere with the colorimetric CN test. Direct distillation of solid samples does not permit testing for sulfide with the spot test. In fact one paper demonstrated that the spot interference tests are not reliable when more than one interference is present. If we used the basic dissolution (in #1) the solution could be treated with Pb and various filtration steps. This is doable but very time consuming. One of the papers claimed that even this may not completely prevent sulfide from showing up in the distillate. Thus, treating the absorber solution with Pb was also necessary, followed by filtration.

My recommendations are to perform our standard water and solid distillation with the following exceptions.

a) use 1g of solid from the air dried and crushed sample prepared for metals analysis. Note in narrative that air drying may result in the loss of HCN if present.

1,3a & 4) Perform the mild basic dissolution on solid samples selected by EPA/BASF. The basic solution would be 0.25M NaOH with 1 mg/mL PbCO<sub>3</sub>. We would use 1g sample plus 50 mL of basic solution. Heat in a boiling water bath for 3 hr. Check to confirm pH >11 after 3 hr. If not

Dave Payne

May 6, 1996

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add NaOH solid and redo. Transfer entire sample and basic solution to distillation tube. Add pH paper. Acidify to  $<2$ . Add usual reagents (be sure to leave 3 minutes between  $H_2SO_4$  addition and  $MgCl_2$  addition). Add 50 mg  $PbCO_3$  or  $CdCO_3$  to absorber solution. Perform normal distillation. Note sample colors at start and end of distillation and include in narrative. Centrifuge absorber solution. Add a drop of  $PbCO_3$  or  $CdCO_3$  solution and observe for  $PbS$  or  $CdS$  precipitate formation. Add Pb solution (up to 10 mL) until no more precipitate forms. Note final volume (if  $>52$  mL) to allow final concentration to be correctly calculated. Centrifuge again. Withdraw a few mL of solution for analysis on the TRAACS.

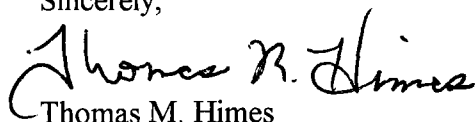
2) Perform all handling of the solid samples in a hood. Special notes should warn analysts about the potential danger of these samples. We should also test the pH of the absorber solution at the end of the distillation. If pH is not  $>12$  then narrate and approach client about re-distillation with more concentrated NaOH in absorber solution. Dilutions of the absorber solution will probably be needed for many samples. All dilutions should be performed with 0.25 M NaOH (not DI water) to keep the pH and dissolved solids content similar to the standards. Prescreening the absorber solutions with CN test strips would also be wise to prevent contaminating the TRAACS.

3b) Perform SCN test on the same samples (different aliquot though) as tested with the special basic dissolution procedure. The SCN test may suffer from other interferences present in the sample matrix but should be useful to the persons interpreting the CN.

I estimate that the special CN distillation and prep would approximately double our cost of performing the CN analysis (not including the SCN test). Also, the particular combination I've recommended above does not appear in any single literature source. All of the changes are similar to Standard Methods 4500-CN, but we do not have any validation data (recovery or MDLs) to support these changes, nor can we guarantee they will be effective on all interferences present at the site.

Once you've had a chance to review the recommendations listed above, please contact Dr. Bruce or myself at (216) 497-9396. Thank you for your consideration and I look forward to your response.

Sincerely,

  
Thomas M. Himes

cc: Bruce Roberts, BASF Corporation  
Kathy Hillig, BASF Corporation  
Mark Bruce, Quanterra Environmental Services

Thomas M. Himes  
Project Manager



Quanterra Incorporated  
4101 Shuffel Drive, NW  
North Canton, Ohio 44720

216 497-9396 Telephone  
216 497-0772 Fax

May 3, 1996

Mr. David Payne  
US EPA  
77 West Jackson Blvd.  
DRT - 14J  
Chicago, IL 60604-3590

Re: BASF RFI Cyanide Studies

Dear David:

Quanterra Environmental Services has conducted a study on potential interferences with the analysis of cyanide by SW 846 method 9012. Two types of interference tests were performed on the midi distillation system.

Thiocyanate

Two method blank distillations were spiked with sodium thiocyanate. Very little cyanide response was measured in the colorimetric determination step. The high (100 mg/L) and low (2 mg/L) level SCN spikes produced CN responses of 3 ug/L and 16 ug/L respectively. Thus, conversion of SCN to CN under the normal conditions in the midi distillation was very low as expected.

Ferricyanide

Three laboratory check samples were spiked with potassium ferricyanide. CN concentration was 42 ug/L. CN recovery was 110, 106 and 103%. Thus, recovery of CN from ferricyanide was complete under the normal midi distillation conditions.

In addition to the above referenced data, please find the enclosed control chart information that summarizes recent cyanide spike recoveries. The improvements made to analytical procedures and reagents has been illustrated by this information.

It is Quanterra Environmental Services' opinion that the midi distillation and method 9012 should be considered acceptable for the analysis of cyanide for the BASF RFI. A letter in reference to alternative methods for samples collected from the Prussian Blue area is being drafted.

Should you have any questions or require further information, please contact myself at (216) 966-9785 or Mark Bruce at (216) 966-7267.

Sincerely,

A handwritten signature in cursive script that reads "Thomas M. Himes".

Thomas M. Himes

Enclosures

cc: Bruce Roberts, BASF  
Mark Bruce, Quanterra Environmental Services



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5  
77 WEST JACKSON BOULEVARD  
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

APR 19 1996

DRT-14J

**MEMORANDUM**

SUBJECT: Environmental Standards, Inc. (ESI)  
SOPs for Data Review/Validation

FROM: David A. Payne, Chemist  
TPS, WPTD

TO: Dianne Sharrow, Environmental Scientist  
Enforcement and Compliance Assurance Branch  
(DRE-8J)

As the dust has settled from the laboratory evaluation of Quanterra Environmental Services, North Canton, Ohio, I have reviewed again data review/validation SOPs of Environmental Standards, Inc. (ESI). The SOPs are equivalent to data review guidelines for EPA's Contract Laboratory Program (CLP) for Target Compound List (TAL) organics. The SOPs do not discuss any specific items for the additional organic compounds necessary for Appendix IX. The SOPs are generally acceptable in concept for data validation.

The ESI SOPs for metals and cyanide should not be significantly effected by extra Appendix IX elements. Appendix IX includes the element tin, but the CLP Target Analyte List (TAL) does not. Tin has poor analytical performance compared to other ICP elements, but I have never known tin to ever be a factor at any RCRA or Superfund site in Region 5. The EST SOPs may not specifically address the ICP Trace instrument, but ESI staff are known to be knowledgeable on its use. When Quanterra completes its work on cyanide, the laboratory should meet, or exceed the criteria established by ESI for acceptable cyanide data. The remainder of this memo will discuss ESI SOPs for organic analysis data. I believe that ESI will not be troubled by the comments below:

ESI should consider, or re-evaluate their SOPs for the following items:

1. The ESI SOPs were written in mid-1995, for use of less than 30% for the % RSD of initial calibration for Methods 8240, 8260, and 8270. SW-846, Update II, now requires less than 15% for initial calibration. I briefly looked at the Method 8260A corporate Quanterra SOP. This specifies 80% of the volatile target compounds shall have an initial calibration with a % RSD less than 15%. This is a unique twist.

2. ESI's SOP for volatiles is written for a 5 ml purge volume, not the 25 ml purge volume of Method 8260. Water miscible volatiles (acetone, acrolein, etc) behave differently as discussed in lab evaluation memo for Quanterra, for 5 ml and 25 ml sample aliquots.
3. ESI's SOPs use a minimum relative response factor (RRF) of 0.05 for volatiles, semi-volatiles and SPCC criteria. Quanterra uses less than 0.3 for volatiles SPCC criteria. My observations have been:
  - a. Poor performance for water miscible volatiles generally has occurred when the target volatiles RRF has been less than 0.05. Certain volatiles (acrolein, acetonitrile, etc.) will never have a RRF greater than 0.05. Acrylonitrile and acetone exhibit borderline behavior. The 0.05 criteria is reasonable.
  - b. The first internal standard used for volatiles (bromochloromethane or pentafluoromethane) can have low response causing a larger RRF than calculated from other internal standards.
  - c. The Quanterra lab evaluation report identified organic compounds that will not, or may not, meet the 0.05 RRF criteria. Four or five Appendix IX organic compounds may not be detectable at all.

The following minor items should be considered by ESI. ESI should have no problems with them.

1. The ESI SOP for sulfide considers sample distillation for sulfide. Quanterra will be separating sulfide by precipitation with zinc hydroxide. The ESI and Quanterra analytical methods are different.
2. The ESI SOPs discuss calibration factors for initial calibrations in Methods 8080 and 8150. Quanterra uses a second order regression for initial calibration of single component compounds in Methods 8080 and 8150. Quanterra uses calibration factors for Aroclors, tech.chlordane, and toxaphene in Method 8080. ESI is familiar with second order regression behavior.

cc: K. Hillig, BASF



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION 5  
77 WEST JACKSON BOULEVARD  
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

**MEMORANDUM**

MAR 6 1996

DRT-14J

**SUBJECT:** Evaluation of Quanterra Environmental Services,  
North Canton, Ohio, for RFI at BASF Corporation,  
Wyandotte, Michigan

**FROM:** David A. Payne, Chemist *D. Payne*  
TPS, Pesticide and Toxics Branch

**TO:** Dianne Sharrow, Ecologist  
Enforcement and Compliance Assurance Branch  
Waste, Pesticides and Toxics Division

**I. INTRODUCTION**

I visited and evaluated Quanterra Environmental Services, North Canton, Ohio during August 1995 for the RCRA Facility Investigation (RFI) to be done at BASF Corporation, Wyandotte, Michigan. The evaluation covered Appendix IX testing of soils and waters, and site-specific propylene oxide and propylene glycol testing. During 1994, I had visited the laboratory to evaluate non-Appendix IX volatiles, gas chromatography, (Methods 8080/8081), metals, and general chemistry. Recommendations for any changes to these tests were resolved by Quanterra, Quanterra's clients, and Region 5 during 1994. The laboratory has an excellent reputation for the organic and metal analyses of Appendix IX.

While time was spent in 1995/96 on applicability, and selection of Quanterra's test procedures to Act 307 requirements, approval/disapproval of Quanterra test procedures is based on Region 5 recommendations for Appendix IX testing. Any discussion of Act 307 will be provided solely as information to BASF, and to Quanterra, with no requirements/recommendations for implementation.

Between August 1995 and present, the Quanterra North Canton, Ohio laboratory has been changing from facility specific SOPs to corporate-wide SOPs that have been written to comply with May 1995 updates to SW-846. The corporate SOPs lack details of the facility SOPs and they provide many options for analytical operations, that are not used by Quanterra's North Canton facility. The October 1995 addendum to the BASF QAPP provided certain corporate SOPs, so that test procedures (metals, Method



8080, Method 8150) have 2 SOPs for each test. Recommendations are made as to acceptability of each corporate SOP. Any recommendations as to non-acceptability are not based on the stringent calibration requirements of SW-846, but on changes to operations, QC audits, reporting limits, etc. The important Quanterra corporate SOPs for Methods 8240, 8260, and 8270 are not yet available for Region 5 review.

The August 1995 visit primarily evaluated the analysis of organic Appendix IX compounds and how these organic compounds are apportioned to different test procedures. Copies of detailed calibration records were obtained for Methods 8240, 8260, 8270, 8080 and 8150 and reviewed in 1995/96 at Region 5's Chicago office. The calibration records contained organic compounds, other than Appendix IX, that could be considered either proprietary, or non-proprietary, depending on the compound. The calibrations were reviewed as to appropriateness and for any mass spectral interferences (Methods 8240, 8260, and 8270). Information was obtained as to applicability and target compounds (Appendix IX and non-Appendix IX) of each test procedure to State of Michigan Act 307 requirements [listed chemical, and required Method Detection Limit (MDL)]. It had been 4 or 5 years since Region 5 had reviewed Quanterra's Appendix IX organic analyses, in detail.

The August 1995 evaluation of Quanterra, North Canton, Ohio reviewed the following analytical methods specified by the RFI QAPP:

- Appendix IX volatiles in water - Method 8260
- Appendix IX volatiles in soil - Method 8240
- Appendix IX pesticides/PCBs (including isodrin, kepone, methyl parathion, and parathion) by Method 8080
- Appendix IX herbicides (including Dinoseb and pentachlorophenol) by Method 8150
- Appendix IX metals and cyanide in soil/water
- Sulfide in soil/water
- Propylene oxide and propylene glycol in soil/water

The metals test procedures include ICP Method 6010, mercury, and GFAA test procedures previously evaluated in 1994. The ICP Trace Method 6010 was new in 1995 and calibration and QC audit data for the ICP Trace instrument was not reviewed in Chicago until 1996.



## **II. SELECTION OF ORGANIC APPENDIX IX COMPOUNDS FOR EACH TEST PROCEDURE**

Quanterra has generally apportioned Appendix IX organic compounds to available test procedures in an acceptable manner. There is no regulatory requirement that a specific Appendix IX compound must be tested by a specific methodology, if 2 or more are available. After 6 or 7 years of Appendix IX testing in Region 5, certain compounds have been found to have such poor analytical performance by traditional method selections, that alternative methods are recommended. Alternative methods are discussed below. The strongest recommendations for change is made for 1,4-dioxane. The reporting of cis 1,2-dichloroethene is important. Other changes are of lesser importance.

### **A. Organophosphorus Pesticides (OPPs)**

Nine (9) OPP compounds that can be tested by Method 8140 and chlorinated dioxin/dibenzofurens have been excluded from the RFI. See QAPP. Quanterra's calibration records indicate 7 of OPP compounds are also tested by Method 8270, with QAPP Table 7-4 specifying 2 of the 7 OPP compounds to be tested by Method 8270. Method 8080 provides for parathion and methyl parathion to be tested and so listed in QAPP Table 7-4. These 9 OPP compounds need not be tested or reported. They can be deleted from the BASF QAPP.

### **B. Volatile Compounds**

1,4 dioxane exhibits abysmal performance by either Method 8240 or 8260, because it has a purging efficiency of 1%, or less at room temperature. A reporting limit of 500  $\mu\text{g/l}$  results.

#### **Recommendation:**

It is strongly recommended that 1,4 dioxane be part of Method 8270. A 10-fold increase in sensitivity will result, versus Method 8240. This recommendation was made in August 1995 to the laboratory. If reporting limits less than 50  $\mu\text{g/l}$  are required for this compound, alternate methods will need to be found.

### **C. Semi-volatiles (Method 8270)**

Three (3) semi-volatile compounds are reported as "not detectable" by most Appendix IX labs. These are:

Aramite  
hexachlorophene  
p.phenylenediamine

The lab can no longer obtain authentic standards for Aramite and proposes not to test, or report it. Aramite is no longer part of Quanterra's calibration standards for Method 8270. Hexachlorophene can not be detected at any concentration. The performance of p.phenylenediamine is so abysmal it is debatable whether it is being detected, or not.

**Recommendations:**

- 1) Aramite and p.phenylenediamine need not be reported, as alternative methods or authentic material are not readily available.
- 2) Change hexachlorophene to Method 8150 as time permits. See Attachment I to this memo. This was discussed with Quanterra in August 1995.

Semi-volatile chlorinated compounds isodrin and kepone are being reported as part of Method 8080. This is appropriate, and should continue as described by QAPP Table 7-4. Three additional semi-volatiles (chlorobenzilate, Diallate, and Pronamide) and Aramite could also be tested by Method 8080, but are listed by Method 8270. We are not recommending any changes from the QAPP proposal. Isodrin and kepone should definitely remain as part of Method 8080.

The QAPP Table 7-4 lists 3 chlorinated herbicides, Dinoseb and pentachlorophenol as part of Method 8150. Dinoseb (2-sec.butyl-4,6-dinitrophenol) and pentachlorophenol are also part of Quanterra's Method 8270.

**Recommendation:**

- 1) Keep pentachlorophenol as part of both Methods 8150 and 8270.
- 2) We note Dinoseb requires changes in Method 8150's extraction solvent. See Quanterra facility SOP. It is optional whether Dinoseb need be tested by Method 8150, if solvent changes degrade analytical performance for remaining herbicide compounds. Dinoseb by Method 8150 is being driven by Act 307 requirements.

Ethyl methacrylate and the 3 dichlorobenzene isomers are being tested by both Methods 8240/8260 and by Method 8270. Calibration standards of each method contain these compounds. Leave this unchanged. One can serve as possible backup to the primary method, if interferences occur.

#### D. NON-APPENDIX IX COMPOUNDS

##### 1) cis 1,2-dichloroethene

This volatile is part of Quanterra's calibration for Method 8240 and 8260. It should be reported with Appendix IX volatiles and be part of QAPP Table 7-4. The cis isomer is the dominant 1,2 dichloroethene isomer found in ground water. Ten years ago, trans isomer data were actually the sum of cis and trans isomers, as the packed GC columns of Method 8240 provided co-elution of the 2 isomers, with equal mass spectra. Region 5 routinely requests the reporting of cis 1,2 dichloroethene with Appendix IX volatiles.

##### 2) a-chlordane and g-chlordane

These individual major components of tech.chlordane are part of Quanterra's routine standards for Method 8080. These should be added to Table 7-4 as part of Method 8080. The two compounds help interpret the presence of any tech.chlordane, a complicated multi-component mixture.

### III. SUMMARY OF FINDINGS AND RECOMMENDATIONS

#### A. ACCEPTABLE TEST PROCEDURES

The following test procedures are acceptable at the BASF subject site:

1. Mercury in soil/water
2. Sulfide in water
3. Extraction of waters and soils for Method 8270 and 8080 (SOP No. CORP-OP-0001). Appendix A to CORP-OP-0001 (Method 8150) is not acceptable.
4. Pesticide/PCBs in soil/water (SOP LM-WALN-4060) is acceptable. SOP CORP-GC-0001 is more generic than facility SOP, and needs the details of the facility SOP.
5. Two (2) Methods 8015 for propylene oxide in soil/water and for propylene glycol in soil/water.
6. Graphite Furnace Atomic Absorption (GFAA) Analysis of soil/water (SOP No. NC-MT-002). GFAA determinations will not be used in initial RFI in 1996. Approval is still recommended for reporting limits specified by this facility SOP. This facility SOP (NC-MT-002) is inappropriate for certain Act 307 requirements, as the instrument parameters, aliquot volumes and standard concentrations would need to be changed to reflect the different reporting limits of Act 307.

## **1. Pesticide/PCB in soil/water (Facility SOP LM-WALN-4060)**

This facility SOP is acceptable in conjunction with corporate SOP No. Corp-OP-0001 for sample preparation. It describes actual operations at Quanterra. Second order regressions are used for initial calibrations of individual compounds, and calibration factors are used for multicomponent materials - Aroclors, Tech.chlordane, and toxaphene. The initial calibrations are used to quantitate sample results so long as continuing calibration standards are valid. We reviewed the second order initial calibration procedure of Quanterra and found it more accurate than the calibration factor procedure in the concentration ranges used for instrument calibration. The facility SOP describes the use of dibutyl chlorendate (DBC) surrogate instead of the decachlorobiphenyl (DCB) surrogate of Quanterra's corporate SOPs. The North Canton Quanterra is using DBC as their Method 8080 surrogate.

The corporate SOP and facility SOP have different lists of target compounds, but no significant difference in reporting limits and initial calibration concentration ranges. The facility SOP is more helpful for data validation than the corporate SOP.

## **2. Propylene Oxide & Propylene Glycol by Method 8015**

The two compounds are tested by separate test procedures. Facility SOP NC-GC-0021 provides for a group of water soluble/miscible volatiles (ketones, alcohols, 1,4 dioxane, etc.). The target list contains both ethylene oxide and propylene oxide. Water aliquots are injected directly to a GC column. Any confirmation of identity is done with a second, dissimilar GC column. This is a routine test procedure at Quanterra. Reporting limits of 1 mg/l result for each target compound. The effectiveness of this test procedure is uncertain, because one never knows the matrix or chromatographic interferences that will occur for the non-specific flame ionization detector (FID) being used.

For propylene glycol (non-volatile), different GC temperature operations and column are needed. As of the last week in February 1996, a single primary GC column was available with no confirmation column yet selected. A reporting limit of 10 mg/l is obtained for propylene glycol. This is undesirable sensitivity of analysis.

The 2 test procedures are acceptable for the RFI at this time. We expect the data will suffer from undesirable sensitivity, matrix effects, and specificity of compound I.D. No easy alternative is available at this time.

## **B. ACCEPTABLE METALS AND CYANIDE TEST PROCEDURES**

On Friday, March 1, 1996 agreement was reached between Quanterra and myself, on the test procedures to use for RFI metals analyses.

### **FINDINGS:**

- 1) Sample digestion procedures using Facility SOPs are acceptable. The QAPP does not contain a corporate SOP for these procedures. Facility SOPs were reviewed at Quanterra prior to 1995.
- 2) Representative soil sample aliquots will be dried, and homogenized (a free flowing powder), prior to selection of analytical aliquots (metals, mercury, cyanide). A facility SOP, used by Quanterra prior to 1996, is available for these sample preparations, and for the QAPP. QC audit results improve with sample homogenization.
- 3) ICP Method 6010A and ICP Trace Method 6010A (Facility SOP NC-MT-0006) would be used for metals in soil and water and would be considered acceptable for the RFI. The ICP Trace instrument is designed to obtain GFAA performance for As, Pb, Se, and Tl. The reporting limits for other ICP Trace elements can be decreased versus ICP Method 6010A.
- 4) The reporting limits (soils) of ICP Method 6010A are usually acceptable for Region 5 RFIs for all elements, except As, Se, and Tl. Cadmium and lead may require increased sensitivity for soils in special cases. This has not been discussed with BASF for Cd and Pb in soil. The Quanterra ICP Trace has specialized measurements for antimony (Sb) versus ICP Method 6010A. The ICP Trace should be more accurate than ICP Method 6010A for Sb.

The reporting limits (waters) for ICP Method 6010A are usually acceptable for Region 5 RFIs for all elements except As, Pb, Se, and Tl, which are done by GFAA sensitivity. The ICP Trace can be used for these 4 elements. The elements Cd, Cr, and Sb sometimes require GFAA sensitivity for non Act 307 waters. These 3 elements should be considered for the ICP Trace instrument.

Region 5 believes there may be a need for lower reporting limits, in the future, for nickel. The current reporting limits of 40 or 50  $\mu\text{g/l}$  may be too high. The 40  $\mu\text{g/l}$  reporting limits are driven by CLP Statement of Work CRDLs. Quanterra could decrease the ICP Trace Reporting Limit from 40  $\mu\text{g/l}$  to 10  $\mu\text{g/l}$  (approx.). Nickel by GFAA can be done with reporting limits of 2-3  $\mu\text{g/l}$ , but this is impossible at

Quanterra, because all GFAA instruments are contaminated with nickel nitrate matrix modifier used for As and Se analyses.

5) During the week of March 4, 1996, discussions with disinterested parties at the Pittsburgh Conference, Chicago, Illinois, indicated the ICP Trace should achieve the reporting limits proposed by Quanterra.

6) Quanterra has not analyzed SRMs (soils) by their ICP Trace, as was done for ICP Method 6010A, 1 to 4 years ago for Region 5.

7) The reporting limits for the Facility SOP should be used for the ICP Trace. The ones that differ from the corporate SOP are:

As	5 $\mu\text{g/l}$	(Fac.)	vs.	10 $\mu\text{g/l}$	(Corp.)
Cd	1	(Fac.)	vs.	2 $\mu\text{g/l}$	(Corp.)
Sb	5	(Fac.)	vs.	10 $\mu\text{g/l}$	(Corp.)

8) Both the facility SOP and corporate SOP are vague on specific operations of the ICP Trace instrument, such as stringent matrix matching, use of internal standards for quantitation, and the specialized background measurements used to correct Sb, Pb, and Se signals. Interelemental corrections also are not the same for the ICP Trace, versus the ICP Method 6010A (1996 Pittsburgh Conference conversations).

#### **Recommendations:**

1) Use SOP NC-MT-006 and associated sample preparations (including homogenization of soils) for metals analyses of soils/waters. Consider this SOP to be an addendum to the more generic SOP CORP-MT-001.

2) Utilize ICP Trace measurements at a minimum for As, Pb, Se, Tl, and Sb in water and As, Se, Tl, and Sb in soil. Sb is preferred by ICP Trace due to accuracy considerations. Cd and Cr ICP Trace measurements are desirable for waters.

Region 5 usually does not require ICP Trace reporting limits for Ag (5  $\mu\text{g/l}$ ). The ICP Trace also utilizes a 1  $\mu\text{g/l}$  reporting limit for copper (Cu). This is optional to Region 5 at this time.

3) BASF and Quanterra should rewrite QAPP Tables reflecting the above considerations and actual test procedures to be used. GFAA need not be included.

4) Review of calibration records for the ICP instruments indicates the ICP Trace "ICSA" QC audit solution has Sb and Cr contamination. This should be corrected.

5) Quanterra should purchase 3-NIST SRM soils - SRMs 2709, 2710, and 2711. These 3 SRMs provide both total metals values and values defined by Method 3050 sample preparation. Quanterra should test these 3 soils, both by ICP Method 6010A and by ICP Trace to demonstrate accuracy of soil measurements. This QA check for accuracy is very desirable.

6) We expect many soils/waters to contain elements at levels significantly larger than ICP Trace reporting limits. ICP Method 6010A is appropriate for these measurements, if used prior to ICP Trace. The ICP Trace instrument should be superior in accuracy for Sb, versus ICP Method 6010A. The Trace should be preferable for Sb.

7) The laboratory uses a cyanide test procedure widely used throughout the country and the Quanterra system. It is the CLP (Inorg) SOW version with midi-distillation. EPA's, Standard Methods', and ASTM's reference method for cyanide is flawed and inherently inaccurate for the Prussian Blue, and even the AOC in QAPP Table 1-3. The laboratory's cyanide test procedure is appropriate for innocuous samples. The laboratory staff does not have knowledge and practice for problem sample types at the site. I will be forwarding, under separate cover, a collection of literature on cyanide analyses.

### **C. TEST PROCEDURES REQUIRING CORRECTIVE ACTIONS**

The following analytical methods will be considered acceptable for the BASF site if the recommendations are implemented:

1. volatile Appendix IX compounds (Methods 8240 and 8260)
2. semi-volatile Appendix IX compounds (Method 8270)
3. Herbicide Appendix IX compounds (Method 8150)

#### **1. Volatile Appendix IX Compounds (Methods 8240 and 8260)**

The two test procedures are quite similar. The same amount of standard, internal standard, surrogate, and matrix spike are added to 5 ml sample aliquot volumes (Method 8240) or 25 ml volumes (Method 8260). Reporting limits established at 5 or 10  $\mu\text{g/l}$  for Method 8240 are decreased 5-fold to 1 or 2  $\mu\text{g/l}$  for well behaved volatiles (ex.-chloroform). Initial calibrations of the instrument are established using two sets of calibration standards - routine volatiles, and remaining volatiles necessary for Appendix IX. The calibration standards also contain non-

Appendix IX compounds (Freon 113, ethyl acetate, MTBE, etc.). Operation of Methods 8240 and 8260, calibrations and continuing calibration updates were found generally acceptable. Review of detailed calibration records indicate problems are present for water miscible volatiles, and for SOPs LM-WALN-3020 and NC-MS-0002.

**FINDINGS AND RECOMMENDATIONS:**

a. The 2 SOPs describe SDWA analytes and not Appendix IX volatiles. Experimental details are not provided for all of the Appendix IX volatiles.

**Recommendations:**

1) Delete and rewrite tables in each SOP so that SOP is specific to Appendix IX. The tables should describe volatile composition and concentration in each of 2 calibration standards, example retention times on the DB-624 or RTX502.2 columns being used, the quant ions and secondary ions used for each volatile, and reporting limits. This has been discussed with Quanterra.

2) The 2 corporate standards should each list any non-Appendix IX volatiles. This is important for data validation and for Act 307. The corporate calibration standards for Appendix IX contain several non-Appendix IX volatiles.

b. The data packages for volatiles contain experimental spectra, background corrected spectra, and a "reference spectra" for comparison. Extracted ion chromatographs are rarely provided. The reference spectra provided by the labs Target data system are either NIST reference spectra, or experimental spectra but most often are NIST reference spectra. The reference spectra are unlabeled as to their source. I obtained this information from extended quant reports for continuing calibration standards. The data validation process for the RFI needs experimental reference spectra generated on the instrument used for analysis. This is not available.

**Recommendation:**

The data validator and the laboratory should discuss the reference spectra to be provided by Quanterra for volatiles, in addition to the NIST spectra currently used for data presentation. The 2 volatile SOPs should discuss this process, as appropriate, in their Sections 11.5.3. The use of NIST spectra alone is inadequate for data validation.

c. The two extended quant reports reviewed for the two Appendix IX "10 µg/l" continuing calibration standards (Instrument A3I503)



demonstrated unacceptable performance for water miscible volatiles. For "good actors" there appeared to be no problem in going from 5 ml aliquots to 25 ml aliquots. Reporting limits for these "good actors" decreased 5-fold. For water miscible volatiles, with poor purging efficiencies, many could not be detected, with acceptable mass spectra, in the continuing calibration standard. Examples of "bad actors" were acetone, methyl ethyl ketone, acrolein, propionitrile, etc. A single corporate calibration standard (stock) appears to be used for both Method 8240 and 8260. This is inappropriate for the water miscible volatiles. This information was provided Quanterra in September 1995 for a RFI site other than BASF.

**Recommendation:**

Quanterra needs to re-evaluate the concentrations of water miscible volatiles in initial calibrations and continuing calibrations both for Method 8240 and for Method 8260. The water miscible volatiles may require different amounts in Method 8240 and Method 8260 to provide acceptable mass spectra throughout the initial calibration range. Quanterra has informally proposed new concentrations for certain volatiles in Methods 8240/8260.

d. Attachment II to this report shows purging efficiencies of water miscible volatiles, as a function of temperature. These data were taken from a U.S. EPA contract publication (late 1980s) for validation of Appendix IX test procedures. Attachment II explains why 1,4 Dioxane is such a "dog" by Method 8260. During my 1995 evaluations of Appendix IX labs, it was apparent that QC criteria of Methods 8240/8260 for initial calibrations (SPCC and %RSD) and continuing calibrations (%RSD and %D) are not applicable to water miscible volatiles with purging efficiencies or analytical performance worse than acrylonitrile. Attachment III lists volatiles in Appendix IX standards at Quanterra with performance worse than acrylonitrile.

**Recommendation:**

Data validation for Methods 8240/8260 needs to assess water miscible volatiles differently than the "good actors". Do not expect quantitative results, but appropriate mass spectra should still be obtained at the reporting limits being used.

e. Mass spectral interferences, or sensitivity problems, were noted for 3 volatiles - acetonitrile, propionitrile, and acrolein. Acetonitrile and allylchloride co-elute on the common DB-624 column used at Quanterra. The 3 mass spectral lines of acetonitrile are contained in, and overwhelmed by the mass spectra of allylchloride. The identification of acetonitrile, if present, is uncertain.

Propionitrile, with abysmal sensitivity for its 2 available mass spectral lines, is sometimes incorrectly assigned to the retention time of chloroprene in calibration standards for the DB-624 columns and sometimes to the retention time of ethyl acetate. The experimental mass spectra presented do not justify the detection of propionitrile at 20  $\mu\text{g/l}$  (Method 8260) or 100  $\mu\text{g/l}$  (Method 8240).

The mass spectral peak areas provided for acrolein at quant ion 56 are too small for compound detection in samples. Continuing calibrations at 20  $\mu\text{g/l}$  or 100  $\mu\text{g/l}$  are used. Response factors and I.D. for acetonitrile and propionitrile are incorrect. These phenomena are noted on the DB-624 column used for most Quanterra volatile analyses. The GC/MS system with a RTX502.2 column did not exhibit the above behavior, as this column resolves the volatiles superior to DB-624. The RTX502.2 column is not as cost effective as the DB-624 systems.

**Recommendations:**

- 1) Separate acetonitrile and allylchloride into separate calibration standards.
- 2) Re-evaluate the calibration standard concentrations/reporting limits used for acrolein and propionitrile.
- 3) Re-evaluate mass spectral interferences of chloropropane and ethyl acetate on propionitrile.

f. The NIST reference spectra presented by Quanterra is incorrect for 1,2-dibromo-3-chloropropane (DBCP). The apparent base peak of 57 is never observed by Methods 8240 or 8260 for DBCP.

**Recommendations:**

- 1) A different, experimental reference spectrum is needed for DBCP. Do not use the NIST spectra.
- 2) It is suggested that a quant ion, other than 39, would be appropriate for DBCP.

g. 40CFR136 and Method 603 specify acrolein is unstable if samples are collected at  $\text{pH} < 4.5$  (acid preserved samples). Informal work at Quanterra (Denver and North Canton facilities) could not demonstrate acrolein is unstable at pH values of 1 to 2. The pH 4.5 requirement is listed in SW-846 for acrolein. I have no definitive recommendations, as the consequences are undesirable by whichever preservation option is used for acrolein. The pH4.5 requirement is a regulatory requirement of 40CFR136 but is not mentioned in Quanterra SOPs.

One other observation is made. The Michigan Act 307 volatile 2-chloroethylvinyl ether disappears in acid solution. This is definite and can be observed in Quanterra's calibration records.

#### **h. Propylene Dichlorides**

The QAPP, for AOC #5, mentions propylene dichloride, or propylene dichlorides. QAPP Comment #D-1 requests TICs associated with PDC be performed as part of volatile analyses. PDC, or propylene dichloride, is 1,2-dichloropropane. To provide better characterization of the PDC spill:

1) Dichloropropane has 4 isomers. Quanterra tests for 1,2-dichloropropane, and has 2 additional isomers in its SDWA volatile standards (2,2DCPA and 1,3DCPA).

2) Dichloropropene has 7 or 8 isomers. Two are in Quanterra's calibration standard (cis & trans 1,3DCPE) and one additional in their SDWA standard (1,1DCPE).

3) Chloropropane has 2 isomers. None are target compounds for Quanterra's test procedures.

4) Chloropropene has 3 isomers. Allylchloride (3-chloropropene) is part of Quanterra's Appendix IX standard but may not be a metabolite of 1,2DCPA.

5) Chloropropyl ethers (semi-volatiles) may be part of this waste. Bis chloroisopropyl ether is a Target Compound for Method 8270.

#### **Recommendations:**

1) If the waste can be characterized as solely, 1,2DCPA, Methods 8240/8260 are sufficient.

2) If the waste is not solely 1,2DCPA, or it is unknown as to the metabolites present (chloropropanes or chloropropenes), a focused TIC approach, with reference to NIST library spectra, would be more helpful than a blind TIC approach. Extracted ion chromatographs could be generated to prove/disprove the presence of suspect volatiles.

3) The approach will depend on BASF's knowledge on the composition of the propylene dichloride waste, or if only TCL or priority pollutant scans were previously done on the waste (only 1,2DCPA will be reported).

4) Authentic standards of chloropropenes, chloropropanes, and dichloropropenes are another alternative to determine reference spectra and retention times.

## 2. Semi-volatile Appendix IX Compounds (Method 8270)

The detailed calibration records for Instrument a4ext3.1 were reviewed for completeness and appropriateness as well as preparation of stock standards. Analytical and calibration performance is impressive, with minimal instrument drift over 5 months. Excellent chromatography and experimental mass spectra were observed.

The laboratory uses 2 sets of standards (TCL and Appendix IX) to calibrate/update their GC/MS system. Besides the routine compounds of Appendix IX, the standard includes certain apparent proprietary compounds and many non-target isomers of target compounds (ex. - 2,3,4,5 tetrachlorophenol is present as well as the Appendix IX 2,3,4,6 tetrachlorophenol).

### FINDINGS AND RECOMMENDATIONS:

a. Method 8270 is acceptable in operation. Quanterra must be complimented for their excellent system. We do have certain minor comments and recommendations.

#### Recommendations:

- 1) The Method 8270 SOP and QAPP Tables 7-4 contain 7 of the 9 excluded OPP compounds. BASF and Quanterra need to address leaving them as part of Method 8270 SOP or deleting them.
- 2) Compound No. 97 of mass spectral library (and corresponding SOP and standard preparation records) is noted as "N-nitro-o-toluidine" (CAS 99-55-8). 5-nitro-o-toluidine is the correct nomenclature to be used for these records and documents.
- 3) 2-Picoline (No. 14) co-elutes (apparently) with "proprietary" compound No. 13. Mass spectral interferences are present if the co-elution is real. Quant ion 93 for picoline is not affected but its remaining spectra is suspect. The quant ion for No. 13 would be effected by picoline. Quanterra needs to resolve this problem.
- 4) The mass spectra and extracted ion chromatographs for p.phenylenediamine are so abysmal, it is difficult to determine whether this is detectable, or not. Quanterra should review the reporting limit used for this compound.
- 5) Hexachlorophene should be deleted from Method 8270, and changed to Method 8150, if it is to be tested.
- 6) Aramite need not be tested.

7) Bis chloroispropyl ether has a doublet GC peak. Quanterra should define whether the relative response factors need be estimated from both peaks, or one peak. This should be added to the SOP under special considerations. This doublet peak is observed at most GC/MS labs, other than Quanterra.

8) Aniline (No. 21) and phenol (No. 22) co-elute. Both compounds have common secondary ions. Aniline, at relatively high concentration, could provide a "false positive" for phenol. The quant ion for aniline is 93 and phenol has 94. The problem for phenol/aniline should be discussed in SOP, with other special problems.

9) Meta and para cresols should be reported as "either/or". Method 8270 can not distinguish them. The same is true for diphenylamine and N-nitrosodiphenylamine.

10) Quanterra uses "Zinophos" as a common, or alternate name for Thionazin (an excluded OPP). See standard preparation records and No. 90 in mass spectral library. The Method 8270 SOP does not mention either Zinophos, or Thionazin. Example Form I "Analysis Data Sheets" from Quanterra contain neither notation. Quanterra has lost Zinophos on reporting forms, possibly due to confusing nomenclature. Thionazin or Zinophos should be reported, if OPPs are to be determined.

11) The secondary ions 75 and 74 are missing from experimental mass spectra of symetrical trinitrobenzene. Quanterra should clarify if this is standard degradation, or should review reporting limits.

12) Attachment IV to this report lists semi-volatile compounds of poor sensitivity, chromatography, or of known extraction problems. Data validation should be done with a knowledge that these semi-volatile compounds will not, or may not meet QC criteria of Method 8270. Results will not be quantitative. Reporting limits will be judgmental. The compounds listed in Attachment IV have also been observed with undesirable performance at Appendix IX labs, other than Quanterra.

### **3. "Herbicide" Appendix IX Compounds (Method 8150)**

The Quanterra corporate SOPs for herbicides were reviewed - Appendix A to CORP-OP-001 and CORP-GC-0001(Method 8150). These are deemed not acceptable at this time. There is an arbitrary 20X dilution of sample extract (documented only by an asterisk on the last SOP page). Target compounds are also different than for facility SOP.

Review of Quanterra's SOP indicates it will get the job done, and provides sufficient detail to be consistent with proposed QAPP Tables.

**Recommendations:**

- 1) Include hexachlorophene as a target compound for Method 8150 (this is not critical, but should be done as soon as practical).
- 2) Decrease the concentrations of surrogate and matrix spikes. They are presently added at the highest initial calibration standard concentration.
- 3) Establish control limits for the test procedure's surrogate spike.
- 4) Maintain pentachlorophenol as a target compound for Method 8150.
- 5) QAPP Table 3-1 has a typo for 2,4-D recovery from soil.

**D. UNACCEPTABLE TEST PROCEDURES**

The following test procedures should be considered unacceptable for the BASF RFI:

1) Sulfide in Soil

The Quanterra SOP does not separate sulfide from soil prior to analytical measurement of sulfide. This comment is routinely provided to Quanterra.

**Recommendation:**

Delete the parameter "sulfide in soil" from the RFI. No one knows how to use the data.

2) Cyanide QC Audits

The QC acceptance criteria provided in Table 3-1 are not acceptable for cyanide.

**Recommendation:**

Evaluate reasons for poor performance, and correct problem.

## 3) Graphite Furnace Atomic Absorption (SOP CORP-MT-0003)

This corporate SOP, reviewed subsequent to the lab evaluation, arbitrarily eliminates method of standard addition quantitations for GFAA metals. RFI QAPP approval should not bog down while we debate this issue. It is moot at moment, since ICP Trace will replace GFAA measurements for the RFI.

## 4) Method 8150 (SOP CORP-GC-0001 and Appendix A to CORP-OP-001)

The corporate SOPs for herbicides arbitrarily dilute the extract 20-fold. This is not apparent when reading the SOP until the last page and an asterisk footnote. Approval of RFI QAPP should not be delayed while we use a DQO process for herbicides - arbitrary 20-fold increase in reporting limits.

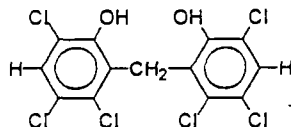
The facility and corporate SOPs have different target compounds.

**Recommendation:**

If reporting limits are ever to be increased for herbicides, a smaller sample aliquot should be used instead of extract dilution. Consider the corporate SOPs not acceptable at this time.

cc: M. DeRosa, ECAB

# Attachment I



Hexachlorophene  
 $C_{13}H_6Cl_6O_2$  Formula Weight 406.92

## Analytical Approach

The first approach to the analysis of hexachlorophene at our laboratory was to use SW846 8270. The recoveries observed using this method were erratic and detection limits were variable. It appeared that some form of chemical degradation or reaction was occurring during the gas chromatographic analysis. Figure 1 illustrates a typical chromatogram of a hexachlorophene standard under the conditions of the 8270 analysis. Not only was the chromatographic peak badly tailing, but the mass spectrum of the peak was not consistent (Figure 2 and Figure 3) with the expected mass spectrum of hexachlorophene. Elevated quantitation limits are often the result of chemical instability and poor chromatography. These limitations of 8270 indicate that an alternative method could lead to better performance.

Figure 1  
Total Ion Chromatogram of 494 mg/L Hexachlorophene

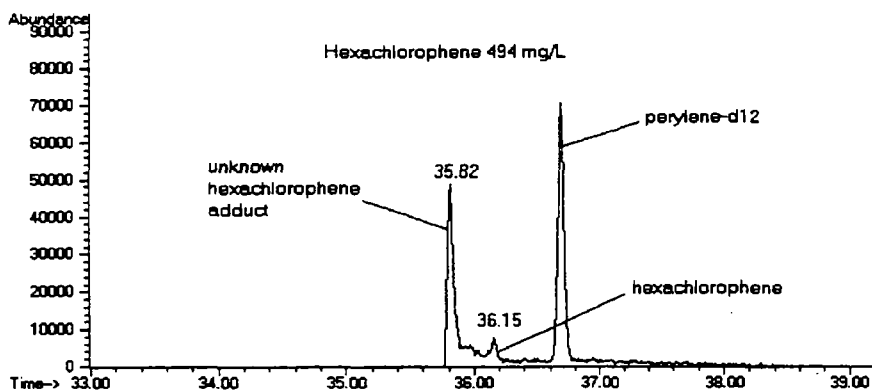


Figure 2  
Mass Spectrum of Unknown Adduct

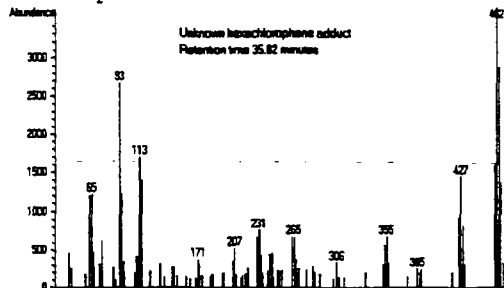
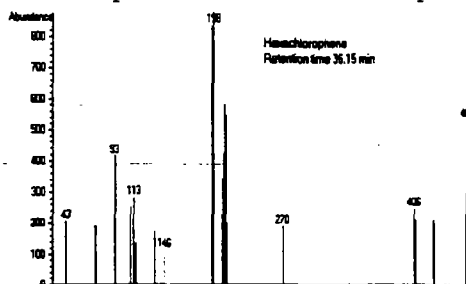


Figure 3  
Mass spectrum of Hexachlorophene



The structure of hexachlorophene indicates that it is a chlorinated phenolic compound that might behave in a similar manner to other

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phenolic compounds such as pentachlorophenol (PCP). PCP is a target compound listed in SW846 8151 Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzoylation Derivatization: Capillary Column Technique. The SW846 8151 method could potentially be applied to the analysis of hexachlorophene. Preliminary mass spectral data (Figure 4 and Figure 5) indicated that complete derivatization was observed when hexachlorophene was methylated with diazomethane.

Figure 4  
Total Ion Chromatogram of  
Methylated Hexachlorophene

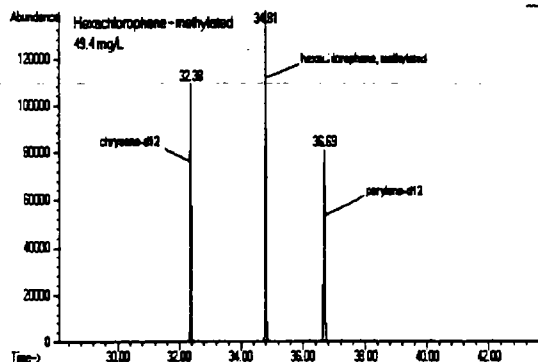
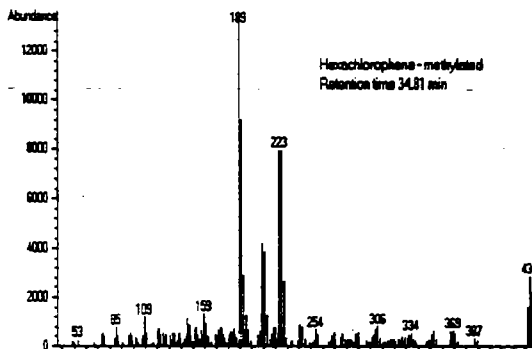


Figure 5  
Mass Spectrum of Methylated  
Hexachlorophene



#### Experimental Design

The first step was to determine the gas chromatographic conditions for the analysis of methylated hexachlorophene. This derivative was not readily available from commercial vendors and so a stock solution of hexachlorophene was derivatized with diazomethane according to the bubbler method described in SW846-8151. From this stock, five levels of calibration standards were prepared at 17.06, 34.11, 68.30, 170.60, and 341.10 ug/l.

The standards were then used to calibrate the gas chromatographic system. The analysis was performed on a HP5890 Series II GC equipped with Electronic Pressure Control and two columns installed into one injection port.

The columns chosen for this analysis were:

Analytical columns:

DB-608, 30 meters, 0.53 mm ID, 0.83 micron film (J&W P/N 125-1730)

DB-1701, 30 meters, 0.53 mm ID, 1.0 micron film (J&W P/N 125-0732)

Guard column:

RTX-5, 3 meters, 0.53 mm ID, 3.0 micron film (Restek Cat # 10282)

The following chromatographic conditions were used:

Injection port- 260 C; Detector- 300 C; Helium carrier at 3.5 PSI; Temperature program- 80 C for 3 min, 5 C/min to 180 C, then 20 C/min to 260 C. Figures 6, 7, 8, and 9 illustrate the chromatographic results and calibration curves that were obtained.

Figure 6  
Chromatogram of Herbicide Methyl Esters and Hexachlorophene on DB-608

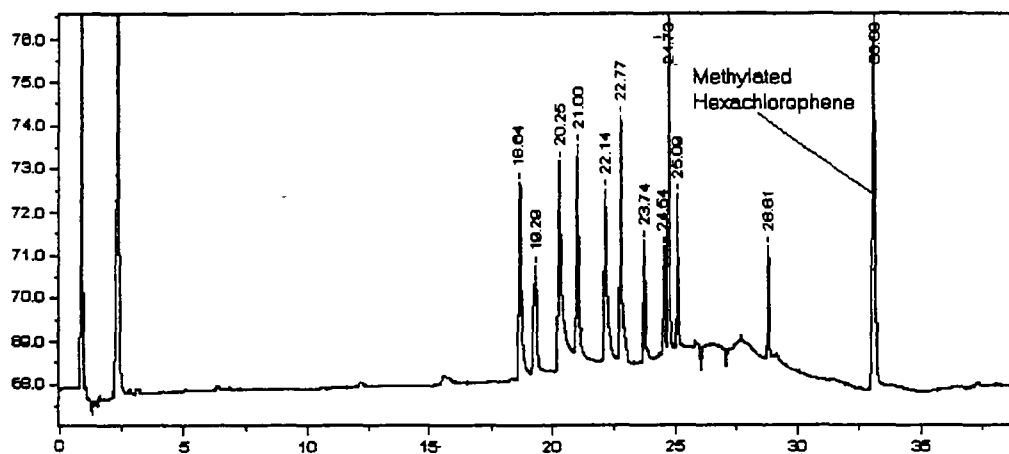


Figure 7  
Chromatogram of Herbicide Methyl Esters and Hexachlorophene on DB-1701

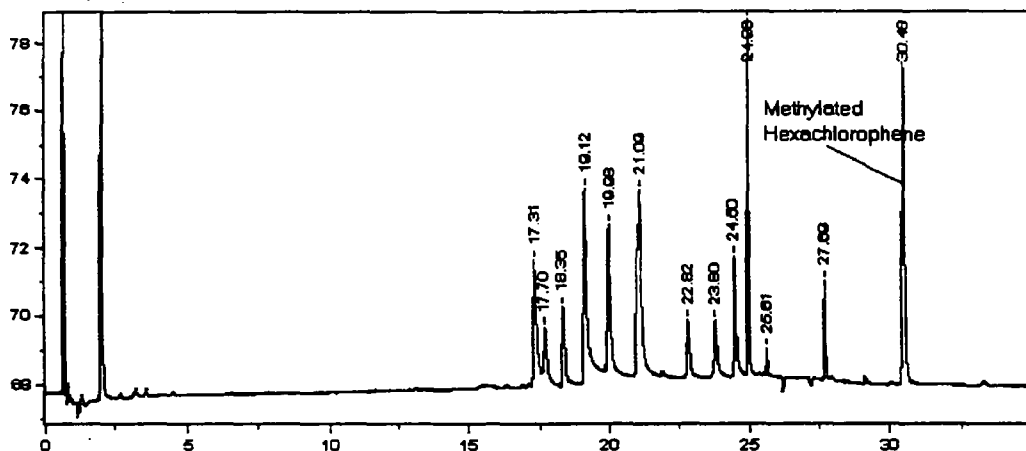


Figure 7  
Calibration Curve on DB-608  
Hexachlorophene Methyl Esters

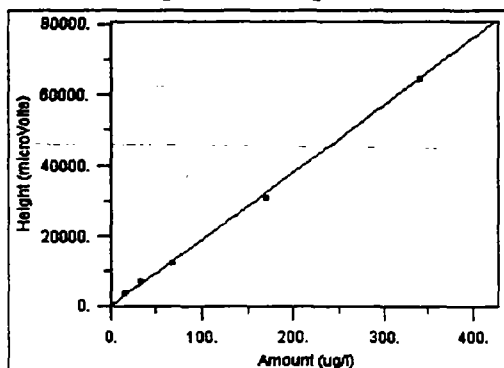
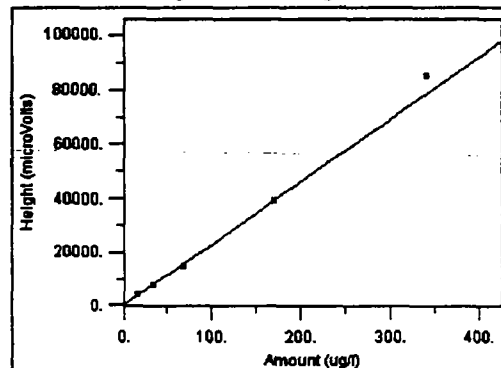


Figure 8  
Calibration Curve on DB-1701  
Hexachlorophene Methyl Esters



The samples were prepared according to the September 1994 revision of SW846-8151 Section 7.0.

The following steps summarize the preparation for waters:

- Add NaCl to 1 liter of sample
- Adjust pH of sample to greater than 12
- Extract with methylene chloride
- Adjust pH of sample to less than 2
- Extract with diethyl ether and dry with sodium sulfate
- Derivatize the extract with diazomethane using the bubbler method

The steps required in the preparation of soils include:

- Adjust pH of sample to less than 2
- Add sodium sulfate
- Extract with methylene chloride/acetone
- Hydrolyze the extract with KOH
- Extract with methylene chloride
- Adjust pH to less than 2
- Extract with diethyl ether and dry with sodium sulfate
- Derivatize the extract with diazomethane using the bubbler method

Preliminary extraction data indicated 40-50% recovery of hexachlorophene through this procedure, and that a significant amount of hexachlorophene was lost in the methylene chloride step. It appears that the pK of the second hydroxyl group is quite high and that even at a pH above 12, the hydrogen is not fully dissociated.

To address the low recovery of the hexachlorophene, the methylene chloride wash step was not performed, but instead, an additional florisil cartridge cleanup was used that was modified from the florisil cleanup described SW846 3620. Preliminary data from real-world soil samples indicate that the florisil cleanup is effective in reducing some types of chromatographic interferences.

A spiked water sample and a spiked soil sample were analyzed in triplicate.

#### Analytical Results

The results of the recovery study are illustrated in Figure 10.

Figure 10  
Recovery Results for Hexachlorophene by SW846-8151  
on DB-608

Sample	Spike amount	Spike found	% Recovery	%RSD
Water 1	9.7 ug/l	7.7 ug/l	79.	
Water 2	9.7 ug/l	8.1 ug/l	84.	
Water 3	9.7 ug/l	9.8 ug/l	101.	
				13.1
Soil 1	320. ug/Kg	240. ug/Kg	76.	
Soil 2	320. ug/Kg	310. ug/Kg	97.	
Soil 3	320. ug/Kg	230. ug/Kg	72.	
Soil 4	320. ug/Kg	270. ug/Kg	84.	
				13.4

Figure 11  
Recovery Results for Hexachlorophene by SW846-8151  
on DB-1701

Sample	Spike amount	Spike found	% Recovery	%RSD
Water 1	9.7 ug/l	9.0 ug/l	93.	
Water 2	9.7 ug/l	11.2 ug/l	115.	
Water 3	9.7 ug/l	12.3 ug/l	127.	
				15.4
Soil 1	320. ug/Kg	280. ug/Kg	87.	
Soil 2	320. ug/Kg	360. ug/Kg	114.	
Soil 3	320. ug/Kg	270. ug/Kg	85.	
Soil 4	320. ug/Kg	300. ug/Kg	95.	
				13.4

#### Conclusion

Recovery data indicate that SW846 8151 can successfully be applied to the analysis of hexachlorophene in soils and waters. This approach results in much improved chromatographic performance. The GC-ECD method provides for more reproducible and reliable detection and quantitation of hexachlorophene than does SW846 8270.

Some work remains to demonstrate the utility of this method and validate its performance. The conditions of the florisil cartridge cleanup need to be finalized. A quad study and method detection limit study need to be performed. The resulting Method Detection Limit and Practical Quantitation Limit obtained using SW846 8151 are expected to be two to three orders of magnitude lower than those obtained using SW846 8270.

**ATTACHMENT III - WATER MISCIBLE VOLATILES WITH PURGING  
EFFICIENCIES AND ANALYTICAL PERFORMANCE  
WORSE THAN ACRYLONITRILE FOR METHOD  
8240/8260**

**APPENDIX IX VOLATILES**

Acetonitrile  
Acrolein  
1,4-Dioxane  
Isobutanol  
Propionitrile

**NON-APPENDIX IX VOLATILES**

n-Butanol  
Cyclohexanone

NOTE: Act 307 specifies MDLs of 1,5, and 10  $\mu\text{g/l}$ , respectively, for acrylonitrile, acrolein, and acetonitrile. These MDLs are inconsistent with Attachments II and III.

**ATTACHMENT IV - APPENDIX IX SEMI-VOLATILES WITH POOR  
SENSITIVITY, EXTRACTION, OR CHROMATOGRAPHY  
BY METHOD 8270 (SPECIAL CONSIDERATION - DATA  
VALIDATION)**

**SEMI-VOLATILES - NO RESPONSE**

Aramite  
Hexachlorophene  
Non-Appendix IX Semi-Volatiles  
(Library Nos. 17, 20, and 147)

**SEMI-VOLATILES - UNDESIRABLE SENSITIVITY**

Dinitrobenzene (3 isomers)  
2,4-Dinitrophenol  
4-Nitrophenol  
4,6-Dinitro-orthocresol  
Pentachloronitrobenzene  
4-Nitroquinoline-1-oxide  
sym.Trinitrobenzene  
Dinoseb  
Famphur  
Semi-volatile #46  
Sulfotepp  
p.phenylenediamine (abysmal chromatography)

**SEMI-VOLATILES - EXTRACTION PROBLEM**

Dimethoate will disappear (hydrolyze) at pH11. If initial extraction is base/neutral, instead of acid/neutral, Dimethoate will have zero percent recovery and should be reported as not detectable.

October 12, 1995

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (HRE-8J)  
77 West Jackson Street  
Chicago, Illinois 60604

RE: Response to EPA's final comments dated August 10, 1995

Dear Ms. Sharrow:

This letter is a response to the another portion of your August 10 comments. BASF will not be able to respond to all of EPA's comments within 60 days as required because of the items to be addressed in the laboratory audit. BASF requests additional time to respond because BASF has not yet received any response from EPA concerning the items being addressed in the laboratory audit.

Below is a brief summary of the revisions to portions of the QAPP as required by (1) your letter dated August 10, 1995, (2) typographical errors, or (3) minor corrections. Again, enclosed are three sets of partial revisions. One set of revisions has the specific revisions highlighted or paragraphs have been bracketed with highlighter and is grouped by EPA comment number except where noted in parenthesis (lab SOPs have not been sequenced by EPA comment and are not highlighted). The other two sets of revisions are in the same sequence as the current QAPP and are not highlighted.

IV. - Two separate Chain of Custody forms will be used. One will be initiated by Quanterra and will be for the empty bottles; it is not enclosed. The second form will be initiated by ESE and will be for the samples (see field SOP-01).

VI.B. - TABLE 6-1 pages 8, 9, and 10 have been revised to specify which methods Quanterra is presently using; the other pages have the correct methods. The North Canton facility is still using Update I methods for several analyses and plans to switch to Update II methods around the end of November. Some of the corporate SOPs reflect Update II methods. When they switch, the tables and lab SOPs will be revised and submitted to EPA for approval. Page 4 has been revised to correct a typographical error. The entire TABLE 6-1 is enclosed. Pages 5 and 6 did not change except for the relative position of the header; the other pages contain revisions.

VI.C. - TABLE 6-1 pages 7, 8, 9, and 10 have been revised to replace the term "RL" with "PQL". Quanterra uses the term "RL" in

their SOPs; the SOPs will not be revised to replace that term. (with VI.B.)

VI.D. - TABLE 1-1, TABLE 3-1, TABLE 6-1 pages 9 and 10, TABLE 7-2, and TABLE 7-4 page 15 have been revised to include the information for propylene glycol and propylene oxide (TABLE 1-1 was submitted with the September 22 response). TABLE 3-1 has also been revised to correct the recovery limits for the ICP metals, correct method revision numbers, add silver (7761) with limits, and add soil pH. TABLE 7-4 page 15 was also revised to correct a typographical error in the units for method 8015A. Quanterra is still in the process of determining the method detection limits for some of the compounds listed in TABLE 7-4; they will be submitted to EPA when they are available. (TABLE 6-1 is with VI.B.)

VII.A. - TABLE 7-1 reflects the methods that Quanterra is presently using; it will be revised when Quanterra switches to the Update II methods. TABLE 7-1 has also been revised to show a SOP for Total Solids, to revise the sequence of the methods, to correct method number revisions, to show the new Quanterra SOP name, to revise note 1, and to add note 3.

VII.B. - TABLE 7-2 reflects the methods that Quanterra is presently using; it will be revised when Quanterra switches to the Update II methods. TABLE 7-2 was also revised by slightly rearranging some of the items, to correct method number revisions, to add soil pH, and to show the new Quanterra SOP name. (with VI.D.)

VII.C. - TABLE 7-4 pages 15 and 17 have been revised to reflect the method Quanterra is presently using. The other pages did not require modification for method revision numbers. Pages 14 and 15 have been revised by shifting the word "method". (all pages with VI.D.)

VIII. - Open issue; to be addressed with lab audit items.

#### MISCELLANEOUS - QAPP

QAPP Section 1 pages 59 through 71 have been respaced to allow the addition of a new section 1.5.20 on page 70 entitled Surface Water Runoff. This new section presents the objectives, tasks, and data usage for collecting surface water runoff samples. The existing Sample Summary Table section has been renumbered to section 1.5.21. Page 2 of 6 of the TABLE OF CONTENTS has been revised to reflect these changes.



XII.A.2. - Quanterra will not have to modify any of their laboratory SOPs for container preservation. SOP-01 Section 3 already states that preservatives will be added. Quanterra will add the preservatives to the bottles before they are shipped. Preservatives would be added in the field if something were to happen when a bottle is being filled or if an empty bottle is broken during transport. As specified in our September 22 response, the pH of preserved samples will be checked in the field and adjusted as required.

When BASF changed from Woodward Clyde to ESE, we did not receive an original copy of some of the forms that appear in the field SOPs. We have recreated most of them and they are enclosed. Their appearance is slightly different but, the content is the same. The enclosed forms are: FIELD BORING LOG - SOP-02 and SOP-19; WELL DEVELOPMENT/REDEVELOPMENT LOG - SOP-07 and SOP-19; SURFACE SOIL DATA COLLECTION FORM - SOP-08 and SOP-19; DRUM FIELD LOG FORM - SOP-12 and SOP-19; DATA SHEET - ELECTRICAL RESISTIVITY SOUNDINGS - SOP-13 and SOP-19; and GROUNDWATER SAMPLE COLLECTION FIELD SHEET - SOP-18 and SOP-19.

SOP-21 has been revised by ESE as discussed with you earlier. It and the cover sheet are enclosed. QAPP Section 1 pages 31 and 32, task 7 have been slightly revised to reflect the modified SOP-21. SOP-21 Section 1.0 - the paragraph beginning "The aquifer testing method...." has been eliminated. The last two sentences in the next paragraph have been replaced with a new sentence and two additional paragraphs have been added. Section 3.1 has been expanded. Section 3.1.1 has been revised by adding two sentences at the end of the second paragraph and adding another paragraph. Section 3.1.2 - the last sentence of the second paragraph has been replaced with three sentences. Section 3.2 has been slightly revised. Section 3.3 has been revised by combining the second and third paragraph and adding an additional paragraph. The word "any" has been added in the second sentence of the last paragraph of the text on page 8.

XIII - The revised and updated laboratory SOPs are enclosed. APPENDIX C cover sheet and Standard Operating Procedures Laboratory Work TABLE OF CONTENTS, without page numbers, have been added. They appeared in the June 1994 QAPP but, not in the March 1995 QAPP. Also, please rearrange the laboratory SOPs so that they match the order shown in the TABLE OF CONTENTS. The enclosed SOPs are already in order.

Also enclosed is TABLE A - COMPARISON OF LABORATORY SOPs. It is for clarification purposes only and is not intended to become part

of the QAPP. It lists the March QAPP SOP name and the QAPP Revision 1 SOP name. The SOPs beginning with "CORP" are corporate SOPs, SOPs beginning with "NC" are newer North Canton SOPs, and SOPs beginning with "LM" are older North Canton SOPs.

XIII.A. - Lab SOPs for propylene glycol and propylene oxide are enclosed with the lab SOPs.

XIII.B. - Section 4.2.16 of the updated Sample Receiving SOP (NC-SC-0005) states that the pHs are taken on all preserved samples except for volatiles and the pH is recorded on the cooler receipt form.

XIII.C.1 - Table 3 in the updated corporate SOP CORP-OP-0001 states that Pest/PCB surrogate is DCB/TCX. Based upon my telephone conversation with you on October 10, it is my understanding that EPA will modify its August 10 comments and drop this comment. Therefore, Quanterra will not modify its SOPs LM-WALN-5020 and LM-WALN-5060 at this time. If EPA does not drop this comment, it will be addressed at a later date.

XIII.C.2. - Audit item.

XIII.C.3. - Audit item.

XIII.D.1. - Audit item.

XIII.D.2 - The lab SOP NC-WC-0004, which includes total solids, has been enclosed with the lab SOPs. The total solids of the sample would be determined by an individual in another section of the laboratory and the results would be entered into the lab's computer system. SOP LN-WALN-5060 does not need to be revised. The computer system would calculate the final results on a dry weight basis.

XIII.D.3. - Audit item.

XIII.E.1. - Audit item.

XIII.E.2. - Audit item.

XIII.E.3. - Comment only; no response is required.

XIII.E.4. - Audit item.

XIII.E.5. - Audit item.

XIII.F.1. - The lab SOP NC-WC-0004 which includes total solids has been enclosed. SOP NC-MT-0005 does not need to be revised; again, the computer system would calculate the final results on a dry weight basis. SOP NC-MT-0005 is in the process of being revised to clarify a reference. When it is revised, BASF will submit the revised SOP to EPA.

XIII.F.2. - Audit item.

XIII.G.1. - Audit item.

XIII.G.2. - Audit item.

XIII.H.1. - Audit item.

XIII.H.2. - Audit item.

XIII.H.3. - The only semivolatile compound for which a TIC search will be used is for aramite; there is no commercial standard available. This was addressed in our September 22 response and is specified in the QAPP Section 1 task 8, page 48 and as a footnote in TABLE 7-4 (QAPP Section 7 page 11).

XIII.H.4. - Lab SOP NC-MS-0004 Table 1 was revised to include aniline and n-nitrosodimethyl amine.

XIII.H.5. - To be resolved with lab audit items.

XIII.H.6. - Audit item.

XIII.I.1. - Audit item.

XIII.I.2. - Audit item.

XIII.I.3. - Audit item.

XIII.I.4. - Lab SOP LM-WALN-4110 section 10.1.15 was revised to state that the snyder column would be prewet with ethyl ether.

XIII.I.5. - Audit item.

XIII.J.1. - Lab SOP LM-WALN-3020 section 10.2.2.2 has been revised to include the length of time of vortexing.

XIII.J.2.- To be resolved with lab audit items.

XIII.J.3. - Audit item.

XIII.J.4. - Lab SOP LM-WALN-3020 section 13.1.1 has been revised to state that the pH of the sample will be checked and recorded.

XIII.J.5. - Audit item.

XIII.J.6. - The only volatile compound for which a TIC search will be used is for isomers of PDC. This was addressed in our September 22 response and is specified in the QAPP Section 1 page 48.

XIII.J.7. - Lab SOP LM-WALN-3020 Table 5 lists methyl ethyl ketone as 2-butanone and does not have to be revised.

XIII.J.8. - To be resolved with lab audit items.

XIII.J.9. - Lab SOP NC-WC-0004 which includes total solids has been enclosed.

XIII.J.10. - Lab SOP LM-WALN-3020 does not need to be revised. The requested information is shown already shown in section 13.2.1.3.

XIII.K.1. - Lab SOP NC-MS-0002 is for aqueous samples. Since there is no need for vortexing, the SOP does not have to be revised.

XIII.K.2. - To be resolved with lab audit items.

XIII.K.3. - Audit item.

XIII.K.4. - Lab SOP NC-MS-0002 section 11.4.1 has been revised to state that the pH of the sample will be checked and recorded.

XIII.K.5. - Audit item.

XIII.K.6. - The only volatile compound for which a TIC search will be used is for isomers of PDC. This was addressed in our September 22 response and is specified in the QAPP Section 1 task 8, page 48.

XIII.K.7. - Lab SOP NC-MS-0002 Table 5 lists methyl ethyl ketone as 2-butanone and does not have to be revised.

XIII.K.8. - To be resolved with lab audit items.

XIII.K.9. - Lab SOP NC-WC-0004 which includes total solids has been enclosed.

XIII.K.10. - Lab SOP NC-MS-0002 does not need to be revised. The requested information is shown already shown in section 11.5.1.4.

XIII.L. - QAPP TABLE 1-3 states that sulfide is a typical coal tar chemical constituent and therefore will still be analyzed for in areas specified in the March 1995 QAPP.

XIII.M. - Audit item.

XIII.N.1. - Quanterra ordered refrigerators in which to store samples and they were delivered on October 9.

XIII.N.2 - Based upon my telephone conversation with you on October 10, it is my understanding that EPA will modify its August 10 comments and drop this comment. Therefore, Quanterra will not modify its SOPs at this time. If EPA does not drop this comment, it will be addressed at a later date.

XIII.N.3. - Quanterra does not routinely confirm "PCBs only" results unless requested by the client; they do confirm the pest/PCB analysis. BASF will request this confirmation.

XIII.N.4. - Audit item.

XIII.N.5. - To be resolved with lab audit items.

XIII.N.6. - Presently, Quanterra is implementing Corporate SOP CORP-GC-0001 which has a TCLP spiking list of Heptachlor, Lindane, Endrin, and Methoxychlor. Refer to Table B-7 in the SOP for spike levels and lists.

XIII.N.7. - SOP NC-OP-0009 GEL PERMEATION CHROMATOGRAPHY CLEANUP and SOP NC-OP-0013 SULFURIC ACID CLEANUP are enclosed.

XIII.O.1. - Audit item.

XIII.O.2. - Audit item.

XIII.O.3. - Audit item.

XIII.O.4. - Audit item.

XIII.O.5. - Audit item.

XIII.P.1. - Audit item.

XIII.P.2. - Audit item.

XIII.P.3. - Audit item.

XIII.P.4. - Audit item.

XIII.P.5. - Audit item.

XIII.P.6. - Audit item,

XIII.Q.1. - SOP NC-QA-0002 Section 4.8 has been revised to state that the bottle is filled. The amount of reagent used depends upon the bottle size. It is Quanterra's policy to check any analyte of interest against the quantitation limit rather than method detection limit.

XIV. - Item 5 on the Chain of Custody Procedures in the Environmental Lab has been revised to state that security is maintained on site. Visitors must be accompanied by a BASF employee; therefore, the possibility of sample tampering is very remote. Item 6 was also revised to state that the sample is either in a secure location or in the custody of an authorized individual. The only test being performed by our Research Services for this RFI is DOT spontaneous combustibility which is not a SW-846 test method.

The Chain of Custody Record has been revised slightly by removing the shading in the RSU# column.

Sincerely yours,



Bruce Roberts  
Project Coordinator

enclosures

cc w/ enclosures:

Ronda Blayer, MDNR-WMD  
Jon Russel, MDNR-ERD  
Bob Veenstra, ESE  
Tom Himes, Quanterra - w/o lab SOPs  
Rock Vitale, ESI - w/o enclosures

**Opal Davis-Johnson**  
**Quality Assurance Manager**



Quanterra Incorporated  
4101 Shuffel Drive, NW  
North Canton, Ohio 44720

216 497-9396 Telephone  
216 497-0772 Fax

August 29, 1995

David A. Payne  
US EPA (SQ-14J)  
77 West Jackson  
Chicago, IL 60604

Dear David:

Attached are the WS and WP results from the last two years you requested per our August 29th phone conversation. Please also find the corresponding corrective action letters.

To answer your question about the vinyl chloride MDLs, the MDLs are from one instrument over several days. The group leader explained that each instrument is used for a particular MDL study such as 5 mL purge, 25 mL purge, SW846 methods vs. 600 series. It is to be hoped that this information will clarify any questions you had about the MDL studies.

If you have any further questions or comments, please feel free to contact me at 216/966-9279.

Sincerely,

A handwritten signature in black ink, appearing to read "Opal Davis-Johnson". The signature is fluid and cursive, with a long horizontal stroke at the end.

Opal Davis-Johnson

Enclosures



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION 5  
77 WEST JACKSON BOULEVARD  
CHICAGO, IL 60604-3590

AUG 10 1995

REPLY TO THE ATTENTION OF:

HRE-8J

**CERTIFIED MAIL P 188 577 128**  
**RETURN RECEIPT REQUESTED**

M W Regional Ecology

AUG 14 1995

Mr. Bruce D. Roberts  
Project Coordinator  
BASF Corporation  
1609 Biddle Avenue  
Wyandotte, Michigan 48192

RE: RFI Workplan Comments  
BASF Corporation, North Works  
MID 064 197 742

Dear Mr. Roberts:

Under Section IX.A.1. of the Administrative Order on Consent (AOC), the United States Environmental Protection Agency (U.S. EPA) has conditionally approved the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) workplan for BASF Corporation, Incorporated's (BASF) North Works Facility. Enclosed you will find U.S. EPA's comments on the RFI Workplan. BASF must revise the RFI Workplan in accordance with U.S. EPA's comments. Per Section IX.A.2. of the AOC, BASF must submit a revised RFI Workplan to U.S. EPA for review and approval, prior to initiating those portions of the RFI affected by these comments. BASF should only submit the revised RFI Workplan pages (including only the revised pages of pertinent attachments, etc.) within sixty (60) days of the certified receipt date of this letter. Submitting only revised pages will expedite the review and approval process, since only the revised pages will need to be reviewed.

As discussed during our meeting on July 21, 1995, the enclosed comments are primarily on the Quality Assurance Project Plan (QAPP). I recognize that some of the comments have already been addressed by BASF. To the extent possible, U.S. EPA's comments have been revised as a result of the meeting and the exchange of documents.

I would also like to take this opportunity to address several "open issues" that both U.S. EPA and BASF agreed to address after the July 21st meeting. Specifically, use of the Michigan Department of Natural Resources (MDNR)





revised "action levels" of June 2, 1995,<sup>1</sup> data validation (qualification of data and 100% data validation), revision of SOP 21 and laboratory audit concerns.

### MDNR ACTION LEVELS

U.S. EPA's Region 5 Office of RCRA (OR) has decided not to allow utilization of the "new" MDNR action levels at RCRA corrective action facilities under a RCRA §3008(h) Order. Although the proposed RCRA Subpart S corrective action rule (Federal action levels) has not been finalized, and specific Federal action levels do not exist, OR believes a site-by-site approach to remediation levels must be taken.

OR is also concerned about the misapplication of Operating Memorandum 14 at corrective action sites under a RCRA §3004 Permit and the validity of the assumptions made by MDNR in preparing Operating Memorandum 14. At this time, the State of Michigan is not authorized to implement the Federal RCRA §3004 corrective action Permit program. Although Michigan has applied for authorization to implement the Federal corrective action Permit program, the application contains the "old" MDNR action levels. Until it has been determined how Michigan intends to utilize the "new" action levels, and whether the "new" action levels affect the implementation of an authorized RCRA §3004 Permit program, OR will not allow the utilization of the "new" action levels at RCRA §3004 corrective action sites.

Despite OR's position, keep in mind that OR does allow for flexibility in determining screening and cleanup levels both on a facility-wide, and unit specific basis, that are reflected in the use and preparation of both human health and environmental/ecological risk assessments (HEA). These assessments allow for assumptions that consider both future and industrial use scenarios.

### DATA VALIDATION

Data Qualification - OR has determined that the data qualification framework proposed by BASF is acceptable. However, BASF must be clear in its submittal of summary data, "how" and "why" the data was qualified based on the proposed framework since a portion of the approach is "unconventional". In addition, the qualifications must be included in the data summaries.

100% Data validation - With few exceptions (facilities with extremely large number of samples, e.g., greater than 1,000), OR requires 100% data validation of all corrective action facilities. Based on the limited number of samples, the distinctness of the SWMUs and AOCs at the North Works, and in an effort to maintain confidence in the data, OR has decided not to allow less than 100% data validation at the North Works.

---

<sup>1</sup> MDNR Operating Memorandum 14, Revision 2 - industrial direct contact values.

SOP 21

Based on the August 3, 1995, conversation between myself and Messrs. Robert Veenstra and Craig Campbell (representatives of ESE), and a review of SOP 21 in conjunction with the Scope of Work, OR agrees with the revisions proposed to SOP 21 by ESE. These revisions should be submitted with BASF's response to the enclosed comments.

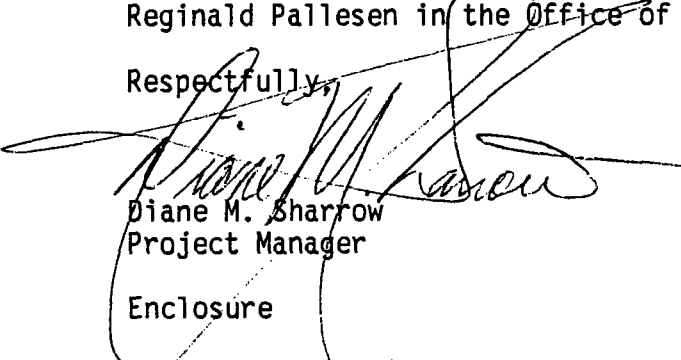
LABORATORY AUDIT

BASF has expressed concern about the number of comments that will be addressed in the laboratory audit of Quanterra to be conducted by U.S. EPA. Specifically, BASF is concerned about delays in RFI implementation if comments are not resolved during the laboratory audit, as well as the timeliness of informing both BASF and the laboratory about the results of the laboratory audit. U.S. EPA is committed to providing to BASF, at a minimum, draft laboratory comments within a few days of the laboratory audit. Concurrently, U.S. EPA will identify what actions must be taken in order to commence field work. It is incumbent upon BASF to follow through with its contracting laboratory (Quanterra) in addressing any issues and concerns, and providing the laboratory with a copy of U.S. EPA's comments.

Lastly, I would like to remind you that BASF must submit ESI's SOPs to U.S. EPA with a claim of business confidentiality under Title 40 of the Code of Federal Regulations (40 CFR 2.203(a)), in order to ensure that U.S. EPA will treat the documents as Confidential Business Information. Information covered by such a claim will be disclosed by U.S. EPA only to the extent and by means of the of the procedures set forth in 40 CFR Part 2, Subpart B. I recommend that both BASF and ESI review these regulations prior to submittal.

If you have any questions or concerns regarding this cover letter or the enclosed comments, do not hesitate to contact me at (312) 886-6199, or Reginald Pallesen in the Office of Regional Counsel at (312) 886-0555.

Respectfully,



Diane M. Sharrow  
Project Manager

Enclosure

cc: BASF Counsel  
R. Blayer, MDNR - Lansing  
L. Aubuchon, MDNR - Livonia  
J. Russell, MDNR - Livonia

## **I. PROJECT DESCRIPTION**

A. Section 1.5.1 (Groundwater Investigation): The phrase "following method in USEPA 1986" is not clear. Revise Task 8 (page 32 of 71) for clarity, to provide the reference for the document.

B. Section 1.5.6 (SWMU E - Polyols Pond): Revise Table 1-1, Section 1.5.6 and Task 2 as follows: For sediment sampling, collect two discrete samples from each pond along the center line of flow. One sample should be at the head end, the other sample at the tail end. All four samples should be sent to the laboratory for the analyses specified in the March 1995 QAPP. Wells RFIMW-1 and 13 should be moved closer to the pond. RFIPZ-2 should be moved south to be utilized for the groundwater extraction system evaluation. Eliminate RFIPZ-1, 3 and 4. Well RFIMW-13 should be analyzed once, and RFIMW-22 should be analyzed quarterly for Appendix IX constituents.

C. Sections 1.5.7 (SWMU F - Filter Cake Disposal Area) and (SWMU G - Two Nominal Rubble Storage Areas): Representative samples of all materials should be analyzed for Appendix IX rather than the TCLP.

D. Section 1.5.9 (SWMU H - Emergency Containment Pond):

1. It is indicated that, from the subsurface screening, the concentration range of the propylene dichloride (PDC) was found to be up to 10,000 ppm in soil. It was not clear whether the reported value was for one of the PDC isomers or for the sum of all of the isomers. BASF should look for TICs associated with 1,2 PDC.

2. Task Number 5 indicated that soil boring will be advanced to a depth of 20 feet. However, it was not clear how samples for the laboratory analysis will be selected from the 20-foot soil column. The QAPP should be clarified to specify sampling strategy and depth.

3. The pond was not lined, and though it was dredged periodically, there is potential for the contaminants to reach groundwater. The entire open drainage system was operated under BASF's NPDES permit. The contaminants that were required to be monitored under the NPDES permit should be included, and it should be stated whether they are on the Appendix IX list.

F. Section 1.5.15 (AOC 6 - Tar Area (South End)): The coal tar area was not lined and there is potential for coal tar constituents to migrate into the surrounding soil as well as groundwater. The boundary of the buried coal tar area must be defined to determine the horizontal and vertical soil contamination and the impact on groundwater. BASF should modify the QAPP by adding a bullet 8 that explains they will move boring activities outward or horizontally if contamination is still found.

G. Section 1.5.16 (AOC 7 - Prussian Blue Area): BASF should analyze for the full Appendix IX list of metals; i.e., all ICP Method 6010 metals, and revise the discussion of this area to clarify the placement of monitoring wells.

## **II. PROJECT ORGANIZATION AND RESPONSIBILITY**

A. ESE should be identified as the party responsible for field collections and field screening and measurements.

## **III. QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY**

A. QAPP Section 3:

1. The method detection limit and/or the sensitivity of the instrument for each analyte of concern should be specified. A reference to Table 7-4 would be acceptable.

2. QC samples should include reagent blanks, field blanks and trip blanks (for VOC analysis only). This section of the QAPP should be modified to reference where this information can be found (Section 8) as well as the frequency of analyzing these QC samples.

3. For the collection of trip blanks for VOC analysis, it should be specified that one trip blank consists of two 40-ml vials.

## **IV. SAMPLING PROCEDURES**

Sample chain-of-custody should be part of the sampling procedure. Chain of custody should be initiated at the time of sample bottle preparation. Bottles prepared at the laboratory should be enumerated.

## **V. CUSTODY PROCEDURES**

A. Section 5.3 (Final Evidence Files): The content of the evidence file (e.g., what type of documents, information and data will be kept in the evidence file?) should be specified, as well as who (e.g., Quanterra) is keeping certain contents.

## **VI. CALIBRATION PROCEDURES AND FREQUENCY**

A. Section 6 should be revised to state that the calibration of the specific conductivity meter and the dissolved oxygen meter will be checked after 10 uses. The field SOPs should also be modified accordingly.

\* B. Table 6-1 should be revised to change method numbers from "8080", "8240A", "8260", "8270A", "8150A", "7470" and "8015" to "8080A", "8240B", "8260A", "8270B", "8150B", and "7470A" respectively, or to specify what methods the laboratory is actually using. The methods do not have to be the latest SW-846 methods.

C. The term "RL" should be changed to "PQL" in all applicable QAPP

tables.

D. All applicable QAPP tables should be revised to include the missing information for the analysis of propylene glycol and propylene oxide.

## **VII. ANALYTICAL PROCEDURES**

A. Some of the method numbers in Table 7-1 should be revised as a result of the update II of the SW-846 methods (arsenic & selenium, change "7060A" to "7060A & 7741A"; semivolatiles, change "3520A" to "3520B"), or to specify the methods the laboratory is actually utilizing.

B. Some of the method numbers in Table 7-2 should be revised as a result of the update II of the SW-846 methods (selenium, change "7740" to "7741A"; pesticides/PCBs, change "8080" to "8080A"; volatile organics, change "8240A" and "8260" to "8240B" and "8260A"; semivolatiles, change "8270A" to "8270B"; herbicides, change "8150A" to "8150B"; propylene glycol/oxide, change "8015" to "8015A"), or to specify the methods the laboratory is actually utilizing.

C. Some of the method numbers in Table 7-4 should be revised as a result of the update II of the SW-846 methods, or to specify the methods the laboratory is actually utilizing.

## **VIII. INTERNAL QUALITY CONTROL CHECKS**

Section 8.1: For analytical results greater than 5 times of the quantitation limit, the RPD should be less than 25% while, for analytical results less than 5 times of the quantitation limit, RPD can be  $\leq 35\%$ . (OPEN ISSUE)

## **IX. DATA REDUCTION, VALIDATION, AND REPORTING**

A. Section 9.2.3: ESI must submit these SOPs to BASF. To insure the handling of these SOPs as Confidential Business Information, BASF must submit them to U.S. EPA as such.

B. Section 9.3: The QAPP should be revised to include calibration check and blank analyses.

## **X. PERFORMANCE AND SYSTEM AUDITS**

Section 10.2.4: Change "Central Regional Laboratory" to "Contract Analytical Services Section of the Monitoring and Quality Assurance Branch."

## **XI. CORRECTIVE ACTION**

A. Section 13.1: The second paragraph of Section 13 should be revised to address corrective action for sampling activities.

## **XII. APPENDIX B - STANDARD OPERATING PROCEDURES FOR FIELD TASKS**

### **A. SOP-01 (Analytical Samples Handling Protocol)**

1. Section 2 (Sample Identification): "Sample Type" is redundant to "Sample Matrix" and should be deleted.

2. Section 3.0 (Sample Containers and Preservation): Quanterra should modify the SOP for container preservation, and a copy should be given to U.S. EPA by BASF.

### **B. SOP-08 (Surface Soil/Sediment Sampling Procedures)**

Section 3.3.4 of SOP 8 should be modified to state that there will be a direct transfer of sample to bottle for volatile analyses.

### **C. SOP-09 (Drilling and Sampling using An Earth-Probe Rig)**

1. Section 5.2, VOCs: SOP 9 should be revised to state that volatile containers will be filled to the top of the container (minimum void).

2. Section 5.3: The SOP heading should be revised by replacing metals with inorganics.

### **D. SOP-10 (Field Measurements of Groundwater Field Parameters)**

The QAPP and field SOP should be revised to add the procedure for continuing calibration check for pH measurement.

### **E. SOP-15 (Pond Sediment Sampling Procedure) and SOP-16 (Surface Water Sampling procedure):**

SOP 8 should be revised to state that there will be a direct transfer of sample to the bottle for volatile analyses.

### **F. SOP-18 (Groundwater sampling):**

SOP 18 should be revised to state that the pH of the preserved sample will be checked and adjusted if necessary.

### **G. SOP-19 (Field Data Recording and Management Procedures):**

The revisions in SOP 18 should be reflected in SOP 19 (record the information of sample preservation in the field logbook and/or the field note sheets).

## **XIII. APPENDIX C - STANDARD OPERATING PROCEDURES FOR LABORATORY TASKS**

A. Analytical methods for the analysis of propylene oxide and propylene glycol were not in the QAPP, but were provided at the July 20, 1995 meeting. Please provide additional copies with your response to these comments.

B. Sample Receiving

Section 4.1.1.6: The SOP must be revised to include checking samples for proper preservation and recording such checks at the time of sample receiving.

C. Continuous Liquid/Liquid Extraction Method for Semi-Volatile Organics in Water Samples

1. The SOP should be revised to include the use of decachlorobiphenyl (dibutyl chlorendate should not be used as a surrogate).

\*2. The matrix spike levels (100/200 ug/L) were too high.

\*3. The concentration level of the LCS (100/200 ug/L) were too high.

D. Sonication Extraction Method for BNAs, Pesticides/PCBs, OPPs, TRPHIRs, and Extractable TPHs in Solid Samples

\*1. Before performing the soil sonication for SVOA7 analysis, at least the pH of the soil needs to be taken. If the pH is above 10, no target acid compounds or surrogates will be recovered from the sample. The pH of the sample should be adjusted with 1:1 HCL to a pH of 7.

\*2. The SOP must be revised for total solids (the method needs to include a procedure for determining the percent moisture; sample results should be reported on a dry weight basis).

\*3. The matrix spike level for BNAs and PAHs were too high.

E. Analysis of Water and Soil Samples for Metals by ICP

\*1. Method only uses a 1-point calibration. RCRA method 6010A requires a 3-point calibration.

\*2. In order to meet the targeted quantitation limits stated in the QAPP, the following metals will have to be measured by GEAA: Antimony, Arsenic, Cadmium, Chromium, Lead, Selenium, Thallium, Zinc and Vanadium.

\* To be addressed in laboratory audit. The results of the laboratory audit will be provided to the facility (BASF) by the Project Manager (D. Sharrow) upon receipt from auditor (D. Payne via M. DeRosa). The facility (BASF) should provide the results to the laboratory (Quanterra).

Addendum to SOP

3. The high pH present in some parts of the site ( $\text{pH} \geq 11$ ) may reduce the probability of finding metals in some samples, since many metals are not soluble in water at this pH.

\*4. The SOP should include the linear range for each metal.

\*5. Method blank should not contain any analyte of interest at concentrations exceeding the method detection limit. When this criteria is exceeded, corrective action should be taken. PQL should be the reporting unit.

F. Analysis of Soil Digestates for Total Mercury by Cold Vapor AA

1. The SOP for total solids should be added (soil results should be reported on a dry-weight basis).

\*2. See also comment XIII.E.5.

G. Total Mercury by Cold Vapor AA for Aqueous Samples

\*1. The analytical procedure presented is acceptable for the analysis of water samples, but is not acceptable for the analysis of soil samples. To prevent thiocyanides and cyanide, which are present at the site, from interfering with the titration, a distillation should be added to the method (See SW846 method 9030A). A copy of the revised SOP that includes the distillation step should be submitted.

\*2. See comment XIII.F.2.

H. GC/MS Semivolatile Organic Compounds/Capillary Column Techniques (Based on Method 8270)

\*1. The percent difference for all compounds in the continuing calibration checks should be  $\leq 30\%$ , except for compounds included in the CCC mix, which should have percent differences  $\leq 20\%$ . The 50% difference in continuing calibration listed in the laboratory method seems excessive.

\*2. The relative standard deviation for all compounds not included in the CCC mix should be  $\leq 15\%$ . For any analyte that does not meet this criteria, a linear equation must be generated for sample quantitation.

\*3. The workplan must be revised to add criteria for performing and reporting tentatively identified compound (TIC) searches.

4. Aniline and n-nitrosodimethyl amine are listed as SVOA analytes for the site, but is not listed as analytes in the SVOA methods. The applicable Qapp table must be revised accordingly.

5. For groundwater samples, the reporting limits for the following compounds are above the targeted quantitation limits listed in Table

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7-4 of the QAPP. A detailed explanation on how the laboratory plans to meet these lower targeted quantitation limits listed in the QAPP needs to be submitted.

1000

→ 1m

or 0.5m/5

<u>Compound</u>	<u>QAPP Target Reporting Limit</u>	<u>Method Reporting Limit</u>
Acenaphthene	5µg/L	10µg/L
Acenaphthylene	5µg/L	10µg/L
Anthracene	5µg/L	10µg/L
Benzo(a)anthracene	5µg/L	10µg/L
Benzo(b)fluoranthene	5µg/L	10µg/L
Benzo(k)fluoranthene	5µg/L	10µg/L
Benzo(ghi)perylene	5µg/L	10µg/L
Benzo(a)pyrene	5µg/L	10µg/L
bis(2-Chloroethyl)ether	5µg/L	10µg/L
bis(2-Ethylhexyl)phthalate	5µg/L	10µg/L
4-Bromophenol phenyl ether	5µg/L	10µg/L
Butyl benzyl phthalate	5µg/L	10µg/L
Chrysene	5µg/L	10µg/L
Diallate	5µg/L	10µg/L
Dibenz(ah)anthracene	5µg/L	10µg/L
Dibenzofuran	5µg/L	10µg/L
di-n-Butylphthalate	5µg/L	10µg/L
Diethylphthalate	5µg/L	10µg/L
Dimethylphthalate	5µg/L	10µg/L
2,4-Dinitrotoluene	5µg/L	10µg/L
2,6-Dinitrotoluene	5µg/L	10µg/L
Fluoranthene	5µg/L	10µg/L
Fluorene	5µg/L	10µg/L
Hexachlorobenzene	5µg/L	10µg/L
Hexachlorobutadiene	5µg/L	10µg/L
Hexachlorocyclopentadiene	5µg/L	10µg/L
Hexachloroethane	5µg/L	10µg/L
Indeno(123-cd)pyrene	5µg/L	10µg/L
Isophorone	10µg/L	20µg/L
Isasafrole	5µg/L	10µg/L
2-Methylnaphthalene	5µg/L	10µg/L
Naphthalene	5µg/L	10µg/L
Nitrobenzene	5µg/L	10µg/L
n-Nitrosodiphenylamine	5µg/L	10µg/L
n-Nitrosodi-n-propylamine	5µg/L	10µg/L
Pentachlorobenzene	5µg/L	10µg/L
Phenanthrene	5µg/L	10µg/L
Pyrene	5µg/L	10µg/L
1,2,4,5-Tetrachlorobenzene	5µg/L	10µg/L
1,2,4-Trichlorobenzene	5µg/L	10µg/L
4-Chloro-3-methylphenol	5µg/L	10µg/L
2-Chlorophenol	5µg/L	10µg/L
2-Methylphenol	5µg/L	10µg/L
2,4-Dichlorophenol	5µg/L	10µg/L
2,4-Dimethylphenol	5µg/L	10µg/L
Phenol	5µg/L	10µg/L
2,4,6-Trichlorophenol	5µg/L	10µg/L

Clean-up

307,

QAPP & SOP

These reporting limits must be revised to match the laboratory SOPs.

\*6. The acceptance criteria specified for phthalates in method blank was unacceptable. The following must be addressed: a) the method blank should not contain phthalate at concentration exceed 5 times of the MDL, not reporting limit, and b) the criteria mentioned above should be applied to only phthalates that are encountered as common laboratory contaminants, not all of the phthalate.

*Lab Audit.*

Herbicides based on Method 8150A/8151 (LN-WALN-4110)

\*1. The volume of the water sample should be measured in a graduate. The practice of assuming a density of one for water samples and weighing 500 g of sample is not acceptable.

\*2. The sample preparation procedure for non-aqueous samples was not acceptable because it was neither the method detailed in Method 8150B nor Method 8151.

\*3. The sample preparation procedure for aqueous samples was not acceptable because it was neither the method detailed in Method 8150B nor Method 8151.

*See Original memo*

4. During solvent concentration, the Snyder column should be prewet with ethyl ether, not acetone or methylene chloride. The SOP should be revised accordingly.

*Type*

\*5. The use of quadratic calibration curves is not acceptable. If a linear fit calibration curve ( $RRF \geq 0.995$ ) can not be drawn, then the instrument should be re-calibrated over a narrower range.

J. GC/MS Volatile Organic Compounds (Method 8240)

1. The SOP must be revised to specify the length of time for vortexing the medium level soil extraction.

2. The minimum RRF for all compounds, except those in SPCC should 0.05. Compounds included in the SPCC should meet the requirements specified in the method. (DATA VALIDATION ISSUE)

\*3. The percent difference for all compounds in the continuing calibration checks should be  $\leq 30\%$ , except for compounds included in the CCC mix, which should have percent differences  $\leq 20\%$ .

4. Because of the high pH associated with some areas of this site, the pH of surface water and groundwater samples should be checked for proper preservation before analysis. Any instance of improper preservation should be noted in the sample results. The SOP must be revised to address this, and submitted accordingly.

\*5. The relative standard deviation for all compounds not included in the CCC mix should be  $\leq 15\%$ . For any analyte that does not meet

*D.P. Feedback*

this criteria, a linear equation must be generated to quantitate the samples.

6. The workplan lists no criteria for performing tentatively identified compound (TIC) searches. See XII.H.3.

7. Methyl ethyl ketone is listed as target analytes in the QAPP, but is not listed in the volatile SOP. The SOP must be revised accordingly.

8. Reporting limits for the following compounds are above the targeted quantitation limits listed in Table 7-4 of the QAPP. The TRLs should be raised to what the laboratory states in the SOPs.

Compound	Method Reporting Limit	Target Reporting Limit
Acrolein	100µg/L	10µg/L
Acrylonitrile	50µg/L	10µg/L
2-Chloro-1,3-butadiene	100µg/L	10µg/L
trans-1,4-Dichloro-2-butene	5µg/L	1µg/L
1,4-Dioxane	1000µg/L	500µg/L
Ethyl methacrylate	100µg/L	10µg/L
Isobutyl alcohol	1000µg/L	500µg/L
Methyl methacrylate	5µg/L	1µg/L
1,4-Dioxane	1000µg/kg	500µg/Kg
Acrylonitrile	50µg/kg	100µg/kg

9. An SOP for total solids should be included (the method needs to include a procedure for taking percent moisture and sample results should be reported on a dry weight basis).

10. The spike level of surrogate compounds used (e.g., addition of x µl of this surrogate standard into 5 ml of sample will yield a concentration of y µg/L.) must be specified.

K. GC/MS Volatile Organic compounds (Method 8260 for Low Level Water)

1. See comments XVII.L.1 - XVII.L.10.

L. Analysis of Sulfide

This SOP must be revised, and is only applicable to AOC 7 (Prussian Blue Area) for sulfides in soil.

M. Cyanide, Automated, Pyridine-Barbituric Acid Method

\*The analytical procedure presented for both water and soil analysis is not acceptable due to the complex nature of the site's sample matrix. Cyanide is present in several species, including ferrous ferric cyanide. The method presented will not accurately quantify the cyanide in all the species present. An alternative procedure is described on page 4-22

of Standard Methods, 18th edition (1992). (OPEN ISSUE)

N. Organochlorine Pesticide/PCBs (Based on Method 8080 and 8081)

1. Sample extracts should be stored at  $4 \pm 2^\circ \text{C}$  to prevent solvent evaporation prior to and after analysis.

2. The use of the surrogate Dibutyl chlorendate (DBC) should be discontinued. Tetrachloro-m-xylene (TCMX) and decachloro-biphenyl (DCBP) should be used as the surrogates for all samples.

3. PCB analysis should be confirmed by a second dissimilar chromatography column. All TICS and NDs should be confirmed.

\*4. Quadratic calibration curves should not be used for analysis. If a linear fit calibration curve ( $\text{RRF} \geq 0.995$ ) can not be drawn, then the instrument should be re-calibrated over a narrower range.

5. For solid samples, the reporting limits for the following compounds are above the targeted quantitation limits listed in Table 7-4 of the QAPP. The limit should be raised to what the laboratory states in the SOPs.

Compound	Method Reporting Limit	Target Reporting limit
alpha-BHC	8 $\mu\text{g/kg}$	1.7 $\mu\text{g/kg}$
beta-BHC	8 $\mu\text{g/kg}$	1.7 $\mu\text{g/kg}$
delta-BHC	8 $\mu\text{g/kg}$	1.7 $\mu\text{g/kg}$
Lindane	8 $\mu\text{g/kg}$	1.7 $\mu\text{g/kg}$
Heptachlor	8 $\mu\text{g/kg}$	1.7 $\mu\text{g/kg}$
Aldrin	8 $\mu\text{g/kg}$	1.7 $\mu\text{g/kg}$
Heptachlor Epoxide	8 $\mu\text{g/kg}$	1.7 $\mu\text{g/kg}$
Endosulfan I	8 $\mu\text{g/kg}$	3.3 $\mu\text{g/kg}$
Dieldrin	16 $\mu\text{g/kg}$	3.3 $\mu\text{g/kg}$
4,4-DDE	16 $\mu\text{g/kg}$	3.3 $\mu\text{g/kg}$
Endrin	16 $\mu\text{g/kg}$	3.3 $\mu\text{g/kg}$
Endosulfan II	16 $\mu\text{g/kg}$	3.3 $\mu\text{g/kg}$
4,4-DDD	16 $\mu\text{g/kg}$	3.3 $\mu\text{g/kg}$
Endrin aldehyde	16 $\mu\text{g/kg}$	3.3 $\mu\text{g/kg}$
Endosulfan sulfate	16 $\mu\text{g/kg}$	3.3 $\mu\text{g/kg}$
4,4-DDT	16 $\mu\text{g/kg}$	3.3 $\mu\text{g/kg}$
Methoxychlor	80 $\mu\text{g/kg}$	50 $\mu\text{g/kg}$
Chlordane	80 $\mu\text{g/kg}$	8.3 $\mu\text{g/kg}$

6. The SOP should be revised to note that the inclusion of heptachlor and chlordane (Technical) in the TCLP spiking solution is not feasible since heptachlor is a constituent of chlordane. Calculating a percent recovery for heptachlor would be impossible.

7. Due to the nature of the site, GPC and sulfur cleanups may

need to be performed on many of the soil samples. A procedure for these two cleanups should be included in the QAPP and the SOP.

## Non-Issue G. Graphite Furnace Analysis

1. Specify the working linear range for each metal of interest.

• \*2. Section 11.2 (Method blank): The following needs to be addressed:

- a. Specifying the frequency of analyzing the method blank;
- b. Specifying the acceptance criteria to be used.

~~Delet~~ c. Method blank should not contain metal of interest at a concentration greater than the method detection limit.

\*3. The analytical spike protocol (Appendix II) was not fully acceptable. When Analytical results of Post-digestion spike samples show the presence of matrix effect, Method of Standard Addition (MSA) should be used and the sample reanalyzed.

\*4. See comment XIII.P concerning the concentration of LCS.

\*5. There will be samples from this site that contain large amounts of oil that might cause broad band absorption on the graphite furnace. Care should be taken to ensure that the samples are completely oxidized.

## No P. Arsenic and Selenium GFAA (Sample) Preparation

\*1. It is not clear whether this SOP was intended for aqueous samples or both the solid and aqueous samples. If the SOP was intended to be used for aqueous, were total or dissolved metals to be determined.

~~SOP~~ \*2. In Section 10.1.4, if it is necessary, sample digestates should be filtered prior to being dilute to volume, and not the other way around.

\*3. In Section 11.2 (Method blank), please address the preparation of method blanks.

\*4. The concentrations for LCS for As and Se specified in Appendix 1 (at 50 ug/l) are unacceptable. The dynamic working linear range for As and Se must be specified in either this SOP or the SOP for GFAA analysis. The laboratory must address whether the LCS at concentration of 50 ug/L will actually fall at the midrange of the linear range for As and Se respectively.

\*5. Using a fixed spiking level (e.g., 50 ug/L) as it was specified in Appendix 1 of this SOP is unacceptable because:

- a. Spike level for sample containing As and/or Se should be

equal to, or slightly above the sample concentration. For samples containing no As and/or Se, the spike level should be 1-5 times of the method detection limit.

b. Spiking at 50 ug/L could bring the total concentration of the spiked samples to exceed the dynamic linear range.

\*6. If aqueous samples containing suspended solid or Solid Samples are to be processed using this SOP, samples need to be homogenized before digestion.

Q. Bottle Blank SOP (NC-OA-002)

Sections 4.8 +

1. The SOP must be revised to 1) specify the volume of reagent water or freon used in each bottle blank preparation, and 2) specify that the bottle blank should not contain any analyte of interest exceeding the method detection limit, not the quantitation limit.

#### **XIV. APPENDIX E - STANDARD OPERATING PROCEDURES FOR RESEARCH SERVICES**

A. The SOP should be revised to insure that the laboratory chain-of-custody procedures include sample tracking during the sample storage, preparation (e.g., extraction, digestion), sample check-in and check out from sample storage, and sample analysis.



8-14-95

To: Charlie Anderson  
Barry Barkel  
Adam Bickel  
Pete Greer  
Kathy Hillig  
Doug Thiel  
Don Yarborough

John Byrnes  
Dale Webster

Bob Veenstra - ESE

Rock Vitale - ESI

Tom Himes - Quanterra

Attached are final comments from EPA concerning the Work Plan. As you can see, EPA conditionally approved the work plan with changes. The nature of many of the changes were worked out at our July meeting. The lab items will hopefully be resolved during the EPA lab audit that is being conducted this week.

I will be making the changes to the QAPP and submitting them to EPA.

*Bruce Roberts*



Setting the Standards for Innovative  
Environmental Solutions

July 21, 1995

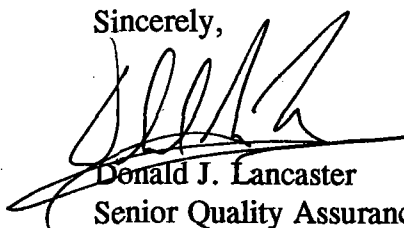
Kathy Hillig, Ph.D.  
BASF Corporation  
1609 Biddle Avenue  
Wyandotte, MI 48192-3799

Dear Dr. Hillig:

Enclosed are the data validation Standard Operating Procedures (SOPs) for the analytical methods to be performed for the Northworks RFI project. The methods include: SW-846 Methods 8240B/8260A (volatiles by gas chromatography/mass spectrometry), Method 8270B (semivolatiles by gas chromatography/mass spectrometry), Method 8080A (pesticides/PCBs), Method 8150B (chlorinated herbicides), Method 8015A (direct aqueous injection volatiles), Method 6010A (metals by ICP), Method 7060A (arsenic by GFAA), Method 7421 (lead by GFAA), Methods 7471A/7470A (mercury by cold vapor AA), Method 7740 (selenium by GFAA), Method 7841 (thallium by GFAA), Method 9012 (cyanide), and Method 9030A (acid-soluble and acid-insoluble sulfides by titration). The data validation SOPs were written specifically for U.S. EPA Region V guidelines and qualifier codes. It should be noted that there is some confusion as to the required method for the analysis of selenium in the project samples. Originally the analysis was to be performed using GFAA (Method 7740); however, a recent communication from EPA stated that the analysis shall be performed using method 7741A, which is a hydride-generation method.

If you have any questions or comments, or if I can be of any further assistance, please feel free to call.

Sincerely,



Donald J. Lancaster  
Senior Quality Assurance Chemist II

DJL:rl

BASF Corporation

**BASF**

July 11, 1995

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (HRE-8J)  
77 West Jackson Street  
Chicago, Illinois 60604

Reference: July 20 meeting

Dear Ms. Sharrow:

The meeting time will be 10:00 AM on Thursday July 20. Tom Himes will not be able to get there any sooner than that.

The purpose of the meeting will be to discuss EPA's draft comments dated June 2, 1995.

The following items are ones that will require an in depth discussion or clarification from EPA.

1. Discussion - Use of a quadratic curve for instrument calibration.
2. Clarification - Method 8270, calibration using calibration standards at concentrations in mg/l range.
3. Clarification - EPA comment VI.D.2.
4. Clarification - EPA comment XII.A.4.
5. Clarification - EPA comment XIII.C.2 & 3.
6. Clarification - EPA comment XIII.K.1.

I believe that the following items were addressed. Please review your comments and we will discuss these at the meeting.

1. EPA comment I.B.1. - The requested information was supplied in the June 1994 Current Conditions Report Appendix H.
2. EPA comment I.B.3.b. - The number of samples is specified in the QAPP on page 38 in Task 2 and in figure 1-8.
3. EPA comment I.B.6.a. - The parameters to be tested are listed in QAPP Table 1-2.
4. EPA comment I.E.1. - Appendix J of the CCR was submitted to EPA last year.

Sincerely yours,

*Bruce Roberts*  
Bruce Roberts

*Copy to: De Rosa  
TSAI*

*File Copy*

15 May 1995

HRE-8J

Ms. Rhonda Blayer  
State of Michigan  
Department of Natural Resources  
Waste Management Division  
Post Office Box 30028  
Lansing, Michigan 48909

- and -

Mr. Jon Russell  
State of Michigan  
Department of Natural Resources  
Southeast Michigan District Office  
38980 Seven Mile Road  
Livonia, Michigan 48152

RE: BASF Corporation, Inc.  
North Works  
Wyandotte, Michigan  
MID 064 197 742

Dear Ms. Blayer and Mr. Russell:

Enclosed you will find a copy of the revised RCRA Facility Investigation (RFI) Workplans and Quality Assurance Project Plans (QAPjP) for BASF Corporation, Inc., North Works in Wyandotte, Michigan. Although the United States Environmental Protection Agency (U.S. EPA), Region 5, is the implementing Agency with regards to the RCRA §3008(h) Administrative Order on Consent, your comments are being solicited due to your expertise and the existence of a State enforcement order also pertaining to the North Works facility. We ask that you pay particular attention to the utilization of Michigan Type C cleanup criteria and methodology. Your comments are appreciated.

I would also like to bring to your attention another U.S. EPA concern regarding the North Works' reporting status in the RCRA Reporting and Information System (RCRIS). Although U.S. EPA continues to enter information into RCRIS and the Corrective Action Reporting System (CARS) module, Michigan is now the implementor of record. Recent pulls from RCRIS have indicated that for the U.S. EPA identification number referenced above, that this facility in Wyandotte is known as Pointe Hennepin, not the North Works. In addition, dates have been entered that do not match activities ongoing as part of this corrective action. Is information from the South Works or the State Order

being entered into RCRIS under this identification number? Do you know who I can contact to ensure that all of the information being entered pertains only to the North Works and whether the North Works has had a name change? Your assistance is appreciated.

U.S. EPA hopes to have comments, and either an approval or approval with comments to BASF Corporation, Inc., by the end of July 1995, depending on the outcome of a laboratory audit of Quanterra, the contract laboratory. If you have any questions or concerns, please contact me at (312) 886-6199. Thank you again for your comments and assistance.

Sincerely,

  
Diane M. Sharrow  
Project Manager

Enclosures (sent to Ms. Blayer)

cc: Bruce Roberts, BASF Corporation, Inc.

April 25, 1995  
4E07014-400

**RECEIVED**  
MAY 19 1995  
**OFFICE OF RCRA**  
WASTE MANAGEMENT DIVISION  
EPA REGION V

Ms. Diane Sharrow  
Project Manager  
U.S. Environmental Protection Agency  
77 West Jackson Blvd. (HRE-8J)  
Chicago, Illinois 60604-3590

Subject: Summary Description of Responses to EPA's Work Plan Comments  
BASF Corporation, Wyandotte, Michigan (Docket No.: V-W-011-94)

Dear Ms. Sharrow:

This letter summarizes revisions to the Work Plans for the RFI at BASF Corporation in Wyandotte, Michigan. This letter responds to your letter dated January 25, 1995.

The responses summarized below describe changes to the work plans. In most cases, we have stated how the plans were reorganized or reconstructed, or where requested information appears.

While the work plans address all comments made by EPA, this summary does not; for example, comments on mislabeled tables or references to "generic" text are not discussed below.

## **GENERAL COMMENTS**

### Project Objectives and Tasks

The project objectives and tasks have been revised to be presented clearly and completely. Overall project objectives and tasks are identified in Section 1.1.1 of the QAPP and discussed in detail in the Sample Network Rationale (Section 1.5).

The QAPP describes only the current Phase of the investigation. This phase will last approximately 13 months, and it will include sampling of soils, installing monitoring wells, performing aquifer tests, quarterly groundwater sampling, reviewing available data and information on the Trenton Channel, and preparing an RFI Report. The QAPP discusses decision points and potential activities (such as sampling and analysis of sediments) that may be included in a subsequent phase of work. The scope of a potential subsequent phase is not discussed in this QAPP, but would be presented to EPA as addenda to the RFI Work Plan.

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Ms. Diane Sharrow  
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## Sample Network Design

Sampling objectives, sampling tasks, and data usages are described in Section 1.5. Rationales are discussed for each objective and task.

The decision process for selecting sample locations is listed where applicable, and the Field Manager will be responsible for making the decisions and notifying the appropriate managers. The sample summary network table was moved into Section 1.0 along with the description of investigative activities. Inconsistencies between the various sections have been addressed.

## Target Parameters and Intended Usage

The analysis to be conducted on samples will be dependent on the wastes that were managed at various areas. For example, samples obtained from the Prussian Blue Area (AOC-7) will be analyzed for RCRA metals and cyanide, because these chemicals were detected there in the past and are the chemicals reasonable expected to be present. The site-wide groundwater investigation and the investigation of background conditions includes analyses for all compounds listed at 40CFR264 Appendix IX except for chlorinated dioxins, chlorinated furans, and organophosphorus pesticides. The rationale for excluding these compounds was discussed with the EPA in October 1994, and is presented in the QAPP.

The sample network design section discusses the tasks that will be performed to investigate the movement of groundwater. The procedures to conduct an aquifer pump test and the data usages also are presented in the QAPP.

## Project Organization and Responsibilities

The responsibilities and/or functions of all relevant project personnel are discussed either in the QAPP or the Project Management Plan. The reference to "Other consultant" in the draft work plan now refers to "contractors", such as drillers and surveyors. BASF will identify the RFI Consultant when selected.

# Woodward-Clyde

Ms. Diane Sharrow  
U.S. Environmental Protection Agency  
April 25, 1995  
Page 3

## SOPs and Analytical Methods

All field and laboratory SOPs are now final versions and have approval signatures. Quanterra will provide SOPs for Method 8015 at a later date.

The SOPs for testing of self-heating materials are presented in Appendix E of the QAPP. The tests will be performed by BASF's Research Services.

An SOP for surface water sampling is included in the event that surface water run-off is found to leave the facility.

Quanterra revised their SOPs to follow SW-846 methods from Update I. Quanterra received Update II information from EPA while preparing the QAPP. Update II can be phased into this RFI over a period of time.

## Document Content and Organization

The QAPP has been reorganized to place all sampling tasks and rationale into Section 1.5. Additionally, many sections were copied from the Current Conditions Report to summarize existing data. Sections 8.0 through 14.0 were revised to be less generic and to be consistent with the model QAPP. We added a reference section (Section 15.0).

## **SPECIFIC COMMENTS**

The title page, table of contents, approval form, and distribution list were revised as requested by EPA.

## Project Description

The QAPP discusses the site-wide groundwater investigation in detail. The investigation now includes a "pumping" test to evaluate the effectiveness of the existing extraction system. Groundwater contours, groundwater gradients, transmissivity, and other physical properties will be measured to describe movement of groundwater and to identify if it is leaving the facility.



## **Woodward-Clyde**

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U.S. Environmental Protection Agency  
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An assessment of sediments in the Trenton Channel will be conducted concurrently with the soil and groundwater investigation at the facility. Initially, the assessment will be a "desk-top" study; any recommendations for additional work would appear in a report. The sediment assessment is described in Section 1.5.19 of the QAPP.

The QAPP describes the target parameters and rationale in Section 1.4. This section also discusses why specific chemicals are excluded from Appendix IX 40CFR264 or why additional chemicals are included.

Both surface and subsurface samples of soil will be obtained during the RFI. In some cases, the former surface of a buried feature is targeted for sampling because it is the most likely location to find contamination, if contamination is present.

The objectives and data usages are explained in the revised work plan for each investigation task. In some cases, the objective of an investigation is to identify only the nature of contamination. Subsequent phases will identify the extent of contamination if contamination is present. All data usages now are consistent with overall project objectives.

The Sampling and Analyses Summary Table (Table 1-1) was revised to show all field and laboratory tests that will be performed.

Section 1.5 has been revised to describe background, objectives, tasks and data usages for each portion of the RFI. Essentially, each part of Section 1.5 discusses an individual SWMU or AOC. Sediments in the Trenton Channel and site-wide groundwater are discussed separately within this section.

Sampling locations will be positioned in the field by referencing figures in the QAPP. The RFI Consultant Field Manager (in consultation with BASF site personnel) will be responsible for staking sample locations.

Background sampling locations for soil and groundwater are now identified in the QAPP. The text explains the rationale for selecting these locations and how data will be processed and evaluated.

DET21975

**Woodward-Clyde Consultants**

# Woodward-Clyde

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The schedule for the RFI is presented in Figure 1-15. We identified a possible delay of seven weeks due to weather or other unforeseen reasons. We did not include this time on the critical path, but we wish to identify this contingency to the EPA.

## Project Organization and Responsibility

This section was revised to discuss the identities and responsibilities of additional individuals. The Project Management Plan discusses this topic in more detail.

BASF has not selected an RFI Consultant, but a selection process was sent to EPA along with the Work Plans.

## Quality Assurance Objectives

The accuracy and precision objectives for all field measurements and devices now are included in the QAPP. Several new tools, such as the geophysical resistivity meter, are added and the QA objectives are described.

The QAPP now discusses completeness, representativeness, and comparability of field and laboratory measurements. The definition of completeness is consistent with the Model QAPP.

Rinsate blanks will be analyzed for the same analytical parameters as the investigative sample. Rinsate blanks will be collected at a frequency of one per day that decontamination activities occur. The numbers in Table 1-1 for these samples are estimates.

One matrix spike/matrix spike duplicate sample pair will be collected and analyzed for every 20 investigative samples. The analyses will be for all parameters identified for the investigative samples.

The table summarizing the sampling and analysis program was Table 3-1 in the draft QAPP; it now is Table 1-1. The table was revised in accordance with comments. The responses are not listed here.

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U.S. Environmental Protection Agency  
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The tables of quality objectives (precision and accuracy) for laboratory analyses were revised extensively to address EPA comments. Please see Table 3-1 and 3-2 for responses.

## Sampling Procedures

The number of samples to be collected at specific areas have been identified and justified. In general, a number of samples was chosen to secure enough data to calculate qualified means and standard derivations and to be consistent with DNR guidelines for sample spacing.

The sample summary table (Table 1-1) was revised to identify the groundwater monitoring wells that will be sampled. Additionally, all wells at the facility will be used to measure groundwater elevations and to construct groundwater contour maps.

Rinsate blanks will be prepared using reagent grade distilled/deionized water. This was agreed upon during the October 1994 meeting with the EPA.

## Custody Procedures

EPA comments on this topic are discussed in Section 5.0 of the QAPP and in field SOP-01.

## Calibration Procedures and Frequency

EPA comments on this topic are addressed in Section 6.0 of the QAPP, in SOP-10 and in SOP-11.

## Analytical and Measurement Procedures

Target reporting limits (TRL) are identified clearly on the summary table (Table 7-4).

Several TRLs are higher than DNR Type B clean-up criteria. The use of Type B criteria is not appropriate at this time because the RFI is an investigation of potential contamination, not a verification of clean-up. Additionally, BASF anticipates pursuing a Type C remedy, not a Type B.

# **Woodward-Clyde**

Ms. Diane Sharrow  
U.S. Environmental Protection Agency  
April 25, 1995  
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## **Sections 8.0 through 14.0**

These sections were revised to present more project-specific information consistent with the Model QAPP.

### **OTHER**

The QAPP discusses tasks to investigate sediments in the Trenton Channel. As suggested in the diagram attached to EPA's comments, BASF will use existing studies to characterize sediment distribution, disposition, quality and sources in the Trenton Channel specifically adjacent to the North Works. At the completion of the work described in the RFI WP, BASF either will document no need for further action or develop a sampling plan. This phased approach to addressing sediments allows for an orderly acquisition of information to assess, investigate and characterize the needs and possible scope for any future work.

### **DESCRIPTION OF CURRENT CONDITIONS**

BASF added Section 3.7 to the Current Conditions Report that summarizes information available on the environmental setting of the Trenton Channel.

### **OTHER REPORTS**

The Pre-investigation Evaluation of Corrective Measure Technologies and the Project Management Plan were revised to include assessment and approach to investigating sediments in the Trenton Channel. These topics were discussed in previous portions of this letter.

The Data Management Plan was not revised because it is not "media-specific". The plan discusses how data are to be managed, regardless of where the data were acquired.

The Health and Safety Plan was not revised to consider contaminated sediment. At this time, we have not proposed locations, methods, or reasons to sample sediments; therefore, it is premature to prepare a safety plan.

## Woodward-Clyde

Ms. Diane Sharrow  
U.S. Environmental Protection Agency  
April 25, 1995  
Page 8

The Public Participation Plan discusses BASF's participation in the RAP and BPAC workgroups.

Sincerely,

A handwritten signature in black ink, appearing to read "John C. Lanigan, Jr.", with a stylized flourish at the end.

John C. Lanigan, Jr.  
Senior Project Geologist

JCL:dcb

cc: B. Roberts  
D. Thiel

DET21975

Woodward-Clyde Consultants

**Woodward-Clyde**

**Woodward-Clyde Consultants**

**Woodward-Clyde**



**Woodward-Clyde Consultants**

March 28, 1995

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (HRE-8J)  
77 West Jackson Street  
Chicago, Illinois 60604

Subject: Submittal of Revised Work Plans  
RCRA Facility Investigation  
Docket No.: V-W-011-94  
BASF Corporation, Wyandotte, Michigan

Dear Ms. Sharrow:

BASF Corporation submits three copies of the revised RFI work plans for the Wyandotte facility in accordance to the schedule specified in your written comments dated January 25, 1995.

The following plans are enclosed:

- Quality Assurance Project Plan
- Current Conditions Report text, some figures and tables, and one page of APPENDIX F EXHIBIT 7;
- Project Management Plan;
- Data Management Plan;
- Health and Safety Plan;
- Public Participation Plan; and
- Corrective Measures Technologies Evaluation.

Also enclosed is our plan to select the RFI Consultant.

Revisions to the QAPP are more extensive than the revisions to the other plans. We believe that these revisions address EPA's concerns.

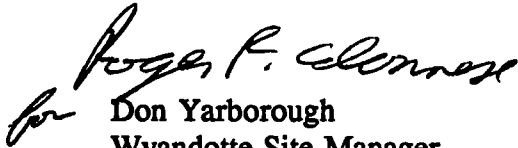
I certify that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to evaluate the information submitted. I certify that the information contained in or accompanying this submittal is true, accurate, and complete. As to those identified portion(s) of this submittal for which I cannot personally



Ms. Diane Sharrow  
United States Environmental Protection Agency  
March 28, 1995  
Page 2

verify the accuracy, I certify that this submittal and all attachments were prepared in accordance with procedures designed to assure that qualified personnel properly gathered and evaluated the information submitted. Based on my inquiry of the person or persons who manage the system, or those directly responsible for gathering the information, or the immediate supervisor of such person(s), the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

Sincerely,

  
Don Yarborough  
Wyandotte Site Manager

Enclosures

cc: B. Roberts - BASF  
J.C. Lanigan, Jr. - WCC



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION 5  
77 WEST JACKSON BOULEVARD  
CHICAGO, IL 60604-3590

**COPY**  
JAN 25 1995

REPLY TO THE ATTENTION OF:

HRE-8J

**CERTIFIED MAIL P 188 577 394**  
**RETURN RECEIPT REQUESTED**

Mr. Bruce D. Roberts  
Project Coordinator  
BASF Corporation  
1609 Biddle Avenue  
Wyandotte, Michigan 48192

RE: RFI Workplan Comments  
BASF Corporation  
North Works  
MID 064 197 742

Dear Mr. Roberts:

Under Section IX.A.1. of the Administrative Order on Consent (AOC), the United States Environmental Protection Agency (U.S. EPA) has disapproved, with comments, the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) workplan for BASF Corporation (BASF). Enclosed you will find the U.S. EPA's comments on the RFI Workplan. BASF must revise the RFI Workplan in accordance with the U.S. EPA's comments. BASF must submit the revised RFI Workplan within sixty (60) days (Section IX.A.2. of the AOC) of the certified receipt date to the U.S. EPA for review and approval. The revised RFI Workplan must indicate where in the text, tables, and various attachments, the U.S. EPA's comments have been addressed.

Though inextricable, the enclosed comments have been divided into two sections; Section I - Quality Assurance Project Plan (QAPjP) comments, and Section II. Other comments. Some comments contain specific examples, but should not be construed as the only specific concerns. Some comments are general, but should not be construed as not needing specific revisions. The comments on the QAPjP were discussed in detail with you and other representatives of BASF on October 17, 1994. The U.S. EPA's sediment concerns were also reviewed in general that day.

The U.S. EPA would also like to take this opportunity to respond to BASF's letters of November 11, 1994, and December 21, 1994, submitted under your signature. With the November letter was enclosed a copy of the report, *Environmental Assessment of Detroit River Sediments and Benthic Macroinvertebrate Communities*, which the U.S. EPA has reviewed. However, the



U.S. EPA does not agree with the conclusion in the letter " . . . that it is [not] necessary to conduct any sediment sampling in front of our [BASF] property.", nor that " . . . any findings would be inconclusive as to the source of the contamination." Please see the enclosed comments for details.

With regards to your December letter, I agree that the letter reflects our telephone conversation for the most part. The changes in the distribution of the Monthly Progress Reports are accurate, and the U.S. EPA's comments on the RFI Workplan are being issued in January. However, I did not commit to a written response by December 23, 1994, on the subject of the specification of a RFI consultant. In our conversation, I stated that I would research the "history" of the requirement and would inform you of the results of my research in writing. I did not specify a date or time-frame for response.

The specification of the RFI consultant in the RFI workplan, is based on U.S. EPA guidance, and as policy has been incorporated into RCRA corrective action guidance, orders and documents. Most importantly, it is a condition in the AOC; Section IX.B. The purpose of this condition is to ensure that the consultant is qualified and will be able to meet the requirements of both BASF and the U.S. EPA. In this case, the consultant must not only be qualified to conduct the corrective action work, but have appropriate sampling and investigative protocols in place to characterize the corrective action necessary at the subject facility.

However, the U.S. EPA is willing to conditionally approve the BASF RFI Workplan, once revised in accordance with the U.S. EPA's comments, without specification of the RFI consultant. The conditions of the U.S. EPA approval would be as follows:

- 1) The RFI Workplan approval would be contingent upon the U.S. EPA's approval of the RFI consultant, (i.e., if the U.S. EPA does not approve of the RFI consultant, the RFI Workplan is not approved and implementation cannot occur).
- 2) BASF cannot utilize selection and approval of the RFI consultant to unduly delay performance, or BASF will be subject to stipulated penalties under Section XV. of the AOC.
- 3) The protocols (e.g., sampling procedures, analytical methods) specified in the RFI Workplan, once conditionally approved by the U.S. EPA, but prior to selection of the RFI consultant, must be followed by the RFI consultant selected by BASF and approved by the U.S. EPA. These protocols will not be subject to revision after the selection of an RFI consultant, or the subsequent approval or disapproval of the RFI consultant by the U.S. EPA.

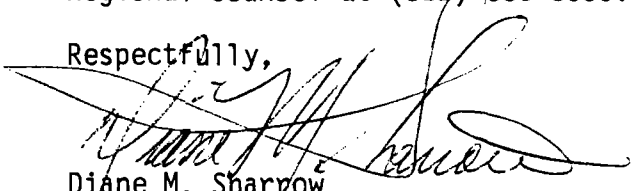
Please provide the U.S. EPA with a detailed plan on how the RFI will be bid and how the RFI consultant will be selected. This information can be provided with BASF's response to the enclosed comments, and this alternative approach should be noted where applicable in all of the plans within the RFI Workplan. The U.S. EPA is particularly interested with the time-frame that will be followed in awarding of the contract. These documents and your response to the conditional approval as outlined above, will be utilized by the U.S. EPA to determine if the conditional approval will be given upon revision of the

RFI workplan in accordance to the enclosed comments.

I do not understand your further statements in the December letter, "... [BASF] would not be able to pick a consultant and revise the plans within 60 days.", and "... [BASF] would not be able to obtain an accurate cost estimate for the work because the work plans will be significantly revised." Section IX.A.2. of the AOC specifies that any submittal should be revised and submitted in accordance with the due date specified by the U.S. EPA. I stated at the October 17, 1994, meeting that a revised RFI Workplan would be due within 60 days of receipt of the U.S. EPA's final comments. If BASF believes that an extension of time is necessary, please submit an extension request for the U.S. EPA's consideration, explaining in detail the circumstances under which an extension is being requested. I believe that the U.S. EPA's flexibility in approving an RFI Workplan without specification of the RFI consultant should alleviate some of the problems that could be encountered in meeting the schedule requirement in the RFI Workplan. If BASF believes the schedules in the RFI Workplan are not achievable, then the schedules should be revised with the submission due within 60 days of the certified receipt of this letter.

Please let me know if BASF is willing to meet the conditions regarding the specification of an RFI consultant. In addition, if you have any questions or concerns regarding this cover letter or the enclosed comments, do not hesitate to contact me at (312) 886-6199, or Reginald Pallesen in the Office of Regional Counsel at (312)-886-0555.

Respectfully,



Diane M. Sharrow  
Project Manager

Enclosure

cc: BASF Counsel  
R. Blayer, MDNR - Lansing  
L. Aubuchon, MDNR - Livonia  
J. Russell, MDNR - Livonia

bcc: Author  
Section File  
Branch File  
R. Pallesen, ORC  
M. Williams, WD - Sediments  
M. Klevs, WD - SEMI  
M. DeRosa, WMD - QAPjP

JAN 25 1995

## **I. QUALITY ASSURANCE PROJECT PLAN (QAPjP)**

The QAPjP was reviewed using the U.S. EPA Region 5 Model RCRA QAPP (Model QAPjP) dated May 1993. The QAPjP contains numerous deficiencies, inaccuracies, and inconsistencies that must be addressed to meet the requirements of the Model QAPjP. In some cases, general comments contain specific examples as an illustration. These examples should not be construed as the only concerns within the given area. Specific comments are also provided after the general comments. Specific comments are presented only once under the section where they first apply; they are not repeated in subsequent sections where they also apply. However, the QAPjP should be revised to address specific comments in all affected sections of the QAPjP and not only in the section where the specific comment is presented. The revised QAPjP should be submitted as Revision 0.

### **A. GENERAL COMMENTS**

This section discusses major QAPjP deficiencies, inaccuracies, and inconsistencies regarding (1) project objectives and tasks, (2) the sample network design, (3) target parameters and intended data usage, (4) project organization and responsibilities, (5) standard operating procedures (SOP) and analytical methods, and (6) document content and organization.

#### **Project Objectives and Tasks**

The QAPjP does not present a clear and complete description of the project tasks associated with overall project objectives, including assessment of past or ongoing releases to the Detroit River. Examples of this deficiency and required revisions are presented below.

1. Overall project objectives are discussed in Section 1.1.1. Some of these objectives are generic and vague. For example, one of the objectives is to relieve threats to human health and the environment from releases of hazardous wastes or constituents from solid waste management units (SWMU) and areas of concern (AOC) at the BASF facility. However, based on the sampling approach and the data usage discussed, it is not clear how threats to human health and the environment will be relieved as a result of the RFI. The QAPjP should be revised to present clear objectives that can be substantiated by the overall sampling and analysis approach. Phases of sampling and analyses may be necessary to address the objective of investigating Detroit River sediment contamination and ecological assessment.

2. The project-specific objectives and associated tasks presented in Section 1.4.1 do not correlate with each other. For example, the project-specific objective for groundwater is to characterize chemicals capable of entering the river or the sewers directly from groundwater discharge. However, the tasks associated with this objective are primarily concerned with the

characterization of the groundwater flow. The QAPjP should be revised to resolve this issue.

3. The QAPjP does not present a clear scope of work with respect to the media being sampled and the types of analyses to be conducted. For example, Section 1.4.3 discusses measurements to be conducted for sediment samples, including physical descriptions, penetration rates, density, weight, and soil organic vapor. However, Section 4.1.4 states that sediment samples from SWMU E will be collected and analyzed for hazardous constituents. In addition, Table 3-1, which summarizes the sampling and analysis program, does not include sampling and analysis of sediment samples. The QAPjP should be revised to summarize all monitoring and screening sampling activities for each media by location as summarized in Table 5 of the Model QAPjP, including Detroit river sediments.

4. Section 1.5.1 states that wells up-gradient from existing contamination will be sampled in order to establish background concentrations of contaminants in groundwater. However, none of the project-specific objectives presented addresses establishment of background concentrations for the contaminants of concern in groundwater or sediment. The QAPjP should be revised to address this issue.

### **Sample Network Design**

The sample network design is vague and confusing. The examples below illustrate deficiencies and inconsistencies of the sample network design.

1. The sample network design components are scattered throughout several sections of the QAPjP. For example, the sample network is discussed briefly in Section 1.5; the sample summary is presented in Table 3-1 of Section 3.0, which discusses quality assurance (QA) objectives; and sampling activities at each SWMU and AOC are presented in Section 4.1.4. Several inconsistencies also exist between Section 4.1.4 and Table 3-1. For example, text in Section 4.1.4 discusses sampling of SWMU E and AOCs 4 and 9; however Table 3-1 does not present any sampling and analysis program for SWMU E or AOCs 4 and 9. The QAPjP should be revised to include a cohesive sample network design with consistent information.

2. Section 1.5.3 states that the general rationale for selecting sampling locations is to examine if contamination in groundwater and soils can potentially enter the Wayne County sewers and the Detroit River. However, no figure is provided that shows Wayne County sewers with respect to the proposed sampling locations. Therefore, the appropriateness of the selected sampling locations cannot be assessed. The QAPjP should be revised to include a figure that shows the Wayne County sewers with respect to the proposed sampling locations, as well as a rationale for sediment sampling locations, and accounts for whether contamination in groundwater and soils has reached the Detroit River in the past.

3. Figure 1-2 shows that the areas of certain AOCs and SWMUs overlap. The QAPjP should discuss the implications of these overlapping areas during sampling and data evaluation.

4. Table 3-1 presents the SWMUs and AOCs to be sampled and the number of soil or groundwater samples to be collected from each SWMU and each AOC. However, no rationale is provided for the selection of the proposed sampling locations. Also, no rationale is provided for the proposed number of samples to be collected from each sampling location. For example, five samples are to be collected from SWMU F. However, the reason for collecting only five samples from this SWMU is not explained. The QAPjP should be revised to provide a rationale for the number of samples to be collected from each SWMU and each AOC. In addition, at a minimum, the Detroit River at current and historic NPDES discharges should be proposed as sampling locations.

5. Table 3-1 does not present the numbers of wells to be sampled or soil and sediment sampling locations for each SWMU, AOC and the Detroit River. For example, Figure 4-3 presents the proposed sampling grid for SWMU F. However, the exact soil sampling locations within the grid are not specified. The QAPjP should be revised to specify the numbers of sampling locations through the combined use of figures and tables.

### **Target Parameters and Intended Data Usage**

Several deficiencies and discrepancies were noted regarding the target parameters and intended data usage. Examples of these deficiencies and discrepancies are presented below.

1. Section 1.4.1 states that groundwater samples will be analyzed for the parameters listed in Appendix IX of 40 Code of Federal Regulations (CFR) Part 264 except for dioxins and furans. However, Table 3-1 does not include organophosphorus pesticides (SW-846 8141) and sulfide (SW-846 9030), which are also parameters included in Appendix IX. The QAPjP should be revised to also include organophosphorus pesticides and sulfide as analytical parameters for this RFI.

2. Text in Section 1.4.1 states that several determinations will be conducted to assess whether groundwater leaves the site or is contained on site. However, the QAPjP does not explain what data will be used or the types of calculations to be conducted to determine if groundwater is leaving the site. The QAPjP should be revised to address these issues.

### **Project Organization and Responsibilities**

Several deficiencies were noted regarding project organization and responsibilities. For example, the responsibilities of several BASF and Enseco-North Canton laboratory personnel presented in Figure 2-2 are not discussed in the text. The QAPjP should be revised to discuss the responsibilities of all personnel presented in Figure 2-2. Also, Figure 2-2 refers to "other consultants" without specifying who the other consultants are. Text in Section 2.0 and the accompanying figures also refer to the "RFI consultant" without identifying who the RFI consultant is. The QAPjP should be revised to identify all consultants to be involved in the RFI, or the approach to be taken with regards to protocols once an RFI consultant is



selected (see the cover letter for additional information).

### **SOPs and Analytical Methods**

Several inconsistencies and deficiencies were noted regarding the field and laboratory SOPs and the proposed analytical methods. Examples of these inconsistencies are summarized below.

1. Field SOPs do not have approval signatures. Also, it is not clear if field SOPs are draft or final SOPs. Only laboratory SOPs LM-WALN-3020, LM-WALN-3040, and LM-WALN-5020 are final and have approval signatures. All other laboratory SOPs do not have approval signatures and are submitted in a draft form. Final versions of the field and laboratory SOPs should be used. Also, approval signatures should be provided for all SOPs.
2. The QAPjP does not provide methods or SOPs to be used for field measurements of pH, temperature, specific conductivity, salinity, turbidity, reduction-oxidation (redox) potential, dissolved oxygen, and water levels/elevations. The QAPjP should be revised to provide SOPs for these measurements.
3. Text in Section 1.4.3 states that physical measurements of sediment and soil samples include physical descriptions, penetration rates, density, weight, and soil organic vapor. However, the QAPjP does not specify the methods or SOPs to be used for these measurements. The QAPjP should be revised to provide SOPs for these measurements.
4. Table 3-1 shows that volatile organic compound (VOC) analysis will be conducted using SW-846 8240. However, the QAPjP includes two laboratory SOPs for VOCs: (1) LM-WALN-3020, which references methods SW-846 8240 and SW-846 8260 and (2) LM-WALN-4180, which references method SW-846 8015. The QAPjP should be revised to address this discrepancy.
5. Section 4.1.4 states that the test method for spontaneous combustion will be conducted in accordance with 49 CFR Part 173 and the SOP presented in Appendix C of the QAPjP. However, Appendix C of the QAPjP does not contain an SOP for spontaneous combustion. The QAPjP should be revised to include an SOP for the spontaneous combustion method.
6. Section 11.1 states that preventive maintenance procedures for field measurements are presented in SOP-02. However, SOP-02 discusses only pH, specific conductivity, and temperature. Because field measurements also include redox potential, dissolved oxygen, salinity, water level elevations, and turbidity, the SOP should be revised to discuss preventive maintenance for the field instruments used for these measurements also.
7. The QAPjP includes SOP-06, which is a field SOP for surface water sampling. However, Table 3-1 indicates that no samples of surface water will be collected. This should be explained and the QAPjP should be revised to include only project-specific SOPs.

8. Table 7-1 summarizes the sample preparation methods to be used. Table 7-1 should be revised to present the SW-846 methods from Update I; therefore, SW-846 methods 3010A, 3020A, 3050A, 3510A, and 3540A should be used instead of SW-846 methods 3010, 3020, 3050, 3510, and 3540, respectively.

9. Table 7-2 summarizes the analytical methods to be used. Table 7-2 should be revised to present the SW-846 methods from Update I; therefore, SW-846 methods 6010A, 8150A, 8240A, 8270A, and 9030A should be used instead of SW-846 methods 6010, 8150, 8240, 8270, and 9030, respectively.

## **Document Content and Organization**

Several deficiencies were noted regarding the QAPjP content and organization. Examples of these deficiencies are summarized below.

1. In general, the QAPjP's organization does not follow the guidelines of the Model QAPjP. For example, Section 1.0 does not summarize existing data to justify sampling locations and parameters for different media to be sampled, including surface water and sediments. Also, the sampling summary table is included in Section 3.0, which presents QA objectives. The description of the SWMUs and AOCs is included in Section 4.0 and not in Section 1.0 as required by the Model QAPjP. The QAPjP should be reorganized to more closely resemble the Model QAPjP in organization and content requirements.

2. The content of Sections 8.0, 9.0, 10.0, 11.0, 13.0, and 14.0 is generic. These sections should be revised to provide project-specific information as required by the Model QAPjP.

3. Several documents are referenced in the QAPjP. However, the QAPjP does not include a list of references. The QAPjP should be revised to include a list of all documents referenced.

## **B. SPECIFIC COMMENTS**

1. The following comments refer to specific sections of the QAPjP. Referenced sections of the QAPjP are identified by section number, page number, and paragraph number where appropriate.

**Title Page** - The title page should be revised to indicate the QAPjP preparer.

**Table of Contents** - The table of contents of the QAPjP should be revised to include lists of figures, tables, and appendixes.

**QAPjP Approval Form** - The QAPjP approval form should be revised to include the following information: (1) the EPA identification number, (2) the revision number, (3) the date of submittal, (4) the QAPjP preparer, and (5) whom the QAPjP was prepared for. The QAPjP approval form should also include Willie Harris as the name of the EPA Region 5 QA manager and specify the name of the RFI consultant. Finally, the QAPjP approval form should be signed by all individuals involved in the QAPjP's preparation except the EPA Region 5

project coordinator and QA manager.

**QAPjP Distribution List** - The QAPjP should be revised to include a QAPjP distribution list, which should identify the names of all individuals receiving copies of the QAPjP.

**Section 1.0, Project Description** - This section and accompanying tables and figures contain numerous deficiencies. Examples of these deficiencies are presented below along with recommended revisions.

1. Section 1.1.3, Page 3, Bullets 1 through 3. This section summarizes the six documents that were used to prepare the QAPjP. Bullet 1 lists "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (QAMS-005/80)" and "Region V Content Requirements for the Preparation of RCRA QAPPs" as two of the documents used for preparing the QAPjP. However, these documents are listed again in Bullets 2 and 3, respectively. Also, the use of so many documents, some of which are more than 10 years old, leads to content and organizational problems. The QAPjP should be revised to follow the organization presented in the Model QAPjP.

2. Section 1.4.1, Page 14, Paragraph 4. The text states that if the data show that groundwater is being contained on site by the existing extraction system, no further investigations will be conducted. This statement is vague, and unacceptable. For example, the text does not explain what the term "data" refers to. Also, the text does not specify the criteria that will be used to decide whether the groundwater is being contained on site. The QAPjP should be revised to explain whether the RFI investigation will be conducted in phases and specify the data and criteria that will be used to decide whether additional phases of investigation are necessary. A concurrent assessment of sediments and ecological risk can be conducted while soil and groundwater is being investigated.

3. Section 1.4.1, Page 14, Paragraph 5. The text states that groundwater samples will be analyzed for parameters listed in Appendix IX of 40 CFR Part 264 except for dioxins and furans. However, the QAPjP does not present any rationale for excluding dioxins and furans from the compounds included in Appendix IX of 40 CFR Part 264. The QAPjP should be revised to provide this rationale.

4. Section 1.4.1, Page 15, Paragraph 1. The text states that "other groundwater samples" will be analyzed for parameters identified for individual SWMUs and AOCs. The QAPjP should be revised to clarify the term "other groundwater samples."

5. Section 1.4.1, Page 15, Paragraph 5. The text states that soil will be sampled at the surface or at the former surface of a buried structure. The QAPjP should be revised to explain why only surface soil samples are to be collected.

The text also states that the groundwater investigation is extensive, with many samples to be analyzed for all chemicals listed in Appendixes IX and 2. However, the text does not specify the parts of 40 CFR that these appendixes

belong to. Also, text in Section 1.4.1, Page 14, states that groundwater samples will be analyzed for the parameters listed in Appendix IX of 40 CFR Part 264 only. This discrepancy should be resolved.

6. Section 1.4.2, Page 16, Bullets 2 and 3. These bullets state that data and information acquired during this RFI will be used to (1) define the nature and extent of chemicals in media to be sampled, (2) evaluate health and environmental risks, and (3) select (an) applicable corrective measure(s). However, it is unclear how the extent of chemicals in media to be sampled will be determined. Also, the intended data usages are inconsistent with the project objectives presented in Section 1.4.1. The QAPjP should be revised to clarify how the extent of chemicals in media to be sampled will be determined and present intended data usages consistent with the project objectives presented in Section 1.4.1.

7. Section 1.4.3, Page 18, Bullets 5 and 6. The text states that surface water and groundwater measurements include pH, temperature, specific conductance, salinity, turbidity, redox potential, and water levels/elevations. However, Table 3-1, which summarizes surface water measurements, does not include water levels/elevations. Table 3-1 also indicates that dissolved oxygen is one of the surface water measurements to be conducted. However, this measurement is not mentioned in Section 1.4.3. The QAPjP should be revised to resolve these inconsistencies.

8. Section 1.4.3, Page 19, Bullet 1. The text states that physical measurements of sediment and soil samples include physical descriptions, penetration rates, density, weight, and soil organic vapor. However, Table 3-1, which presents the sampling and analysis program, does not present any of these measurements. The QAPjP should be revised to resolve this inconsistency.

9. Section 1.4.3, Page 19, Item No. 3. The text states that laboratory SOP names and their equivalent U.S. EPA preparation are presented in Table 7-9. However, Table 7-9 presents laboratory detection limits for toxicity characteristic leaching procedure (TCLP) parameters. The QAPjP should be revised to address this inconsistency.

10. Section 1.4.3, Page 20, Bullets 1 and 2. The text states that laboratory SOP names and their U.S. EPA equivalents for soil and groundwater sample analyses are presented in Table 7-2. However, Table 7-2 presents the U.S. EPA-approved methods, but not the SOP names. The QAPjP should be revised to address this inconsistency.

11. Section 1.5, Pages 20 and 21. The content of this section does not address the sample network, design, and rationale. The text is generic and does not present the selected sampling locations (and especially depths) chosen in conjunction with each SWMU and AOC. This section also does not present the number of samples to be collected from each sampling location, along with a statistical basis supporting the number of samples to be collected. The QAPjP should be revised to include the required information specified in Section 1 of the Model QAPjP.

12. Section 1.5.1, Page 22, Paragraph 1. The text states that soil sampling locations will be marked in the field by the sampling team with a painted wooden stake. Also, the text states that after sampling, the locations will be marked with flags so that they can be located by survey. However, the text does not explain how the exact sampling locations will be chosen and marked before sample collection. The text should be revised to address this issue, as well as sediment sampling.

13. Section 1.5.1, Page 23, Paragraph 1. The text states that wells up-gradient from existing contamination will be sampled in order to establish background concentrations for groundwater migrating to the BASF facility. However, the text does not specify the wells to be sampled. The text also does not explain how many samples will be collected and how the analytical results will be processed to determine background concentrations. This information should be provided.

14. Section 1.5.3, Page 23. The content of this section is generic because it does not identify the sampling locations and rationale for their selection. The QAPjP should be revised to provide this information.

15. Section 1.5.4, Page 23. The text states that the sample summary is presented in Tables 3-1 through 3-13. However, the sample summary is presented only in Table 3-1. The QAPjP should be revised to address this discrepancy.

16. Section 1.6, Page 24. The text states that Figure 1-7 presents the schedule anticipated for the RFI. Figure 1-7 shows that a period of possibly 3 months of downtime is anticipated due to weather conditions after sampling and analysis is conducted. The QAPjP should be revised to clarify how the downtime due to weather conditions after sampling and analysis will impact the overall project schedule.

**Section 2.0, Project Organization and Responsibility** - This section and accompanying figures contain numerous deficiencies. This section generally discusses management responsibilities, quality assurance responsibilities, laboratory responsibilities, and field responsibilities. However, the text does not discuss the responsibilities of all the parties involved in this project. For example, the responsibilities of U.S. EPA Region 5 are not discussed. Examples of specific deficiencies are presented below, along with some recommended revisions.

1. Section 2.1, Page 1. The text states that an overall project organization chart is presented in Figure 2-1 and that the RFI project team organization chart is presented on Figure 2-2. However, several individuals shown in these figures and their responsibilities are not discussed in the text. For example, the U.S. EPA Region 5 project coordinator, D. Sharrow, is never mentioned in the text, and her responsibilities are not discussed. Also, the QAPjP approval form shows that Ms. Sharrow is the U.S. EPA Region 5 project manager and not project coordinator. In addition, the following BASF personnel shown in Figure 2-2 are not discussed in the text: D. Thiel, J. Byrnes, K. Hillig, C. Anderson, B. Barkel, D. Martin, and D. Webster. The

text should be revised to consistently discuss the responsibilities of all personnel presented in the figures.

2. Figure 2-2 shows that T. Himes represents the Enseco-North Canton laboratory. However, the QAPjP approval form shows that Mr. Himes is the project manager of the Enseco-Wadsworth/Alert laboratories. The QAPjP should be revised to resolve this inconsistency.

3. Section 2.2, Pages 1 and 2. This section discusses project management responsibilities of the RFI consultant and BASF. However, the text does not identify the RFI consultant and the RFI project manager. The QAPjP should be revised to identify the RFI consultant and the RFI project manager, or the approach to be followed in selection of the RFI consultant. The QAPjP should also discuss the U.S. EPA U.S. Region 5 management responsibilities.

4. Section 2.3, Page 3. This section discusses the responsibilities of the quality assurance officer (QAO). However, the text does not specify if this title refers to the BASF, RFI contractor, laboratory, or U.S. EPA Region 5 QAO. Also, Figure 2-2 identifies K. Hillig as the BASF QAO. However, the responsibilities of K. Hillig are not identified in this section. The QAPjP should be revised to identify the QAOs of the RFI contractor, laboratory, and the U.S. EPA Region 5, and discuss their responsibilities, as well as those of K. Hillig.

5. Section 2.4, Pages 3 and 4. The content of this section is generic and contains major deficiencies. For example, the location of the laboratory is not specified, analytes and matrices to be tested by the laboratory are not specified, and the text discusses the responsibilities of the laboratory quality assurance/quality control (QA/QC) manager but does not identify any other laboratory staff project responsibilities such as the laboratory project manager and the sample custodian. The QAPjP should be revised to address these issues.

**Section 3.0, Quality Assurance Objectives** - This section and accompanying tables and figures contain numerous deficiencies. Examples of these deficiencies are presented below, along with some recommended revisions.

1. Section 3.1.2, Page 2. This section discusses field precision objectives for pH, specific conductivity, temperature, and redox potential. However, this section does not discuss precision objectives for dissolved oxygen and turbidity, which are also field measurements. The QAPjP should be revised to discuss precision objectives for dissolved oxygen and turbidity.

The text also states that precision goals for pH measurement for replicate samples are  $\pm 0.1$  standard pH units. The text also states that precision goals for specific conductivity are  $\pm 20$  micromhos per centimeter. This statement implies that precision for pH and specific conductivity will be assessed by determining the absolute difference of duplicate pH measurements. However, the text also states that the precision of pH will be assessed through replicate measurements with a maximum relative percent difference (RPD) of 3 and that the precision of specific conductivity will be assessed

through replicate measurements with a maximum RPD of 20. The QAPP should be revised to resolve these discrepancies.

2. Section 3.2.2, Page 3. This section discusses field accuracy objectives for pH, specific conductivity, and temperature. However, this section does not discuss accuracy objectives for redox potential, dissolved oxygen, and turbidity, which are also field measurements. The QAPjP should be revised to discuss accuracy objectives for redox potential, dissolved oxygen, and turbidity.

The text also states that for pH, the accuracy goal is that calibration measurements must be within  $\pm 0.1$  standard pH units for the buffer solution values. The text also states that for specific conductivity, the accuracy goal is that calibration measurements must be within  $\pm 20$  micromhos per centimeter of the true value of the calibration solution. The QAPjP should be revised to indicate that for determining the accuracy of pH measurements, a buffer solution different from the one used for calibrating the pH meter should be used. The QAPjP should also indicate that for determining the accuracy of specific conductivity measurements a standard solution different from the one used for calibrating the specific conductivity meter should be used.

3. Section 3.2.2, Page 3, Paragraph 3. The text states that the accuracy of temperature readings will be evaluated by performing post-measurement verifications. However, the text does not explain how the post-measurement verifications will be conducted to determine accuracy. The text also does not present the accuracy objectives for temperature. The QAPjP should be revised to address these issues.

4. Section 3.3, Page 4. The text discusses laboratory completeness for the samples analyzed. The text should be revised to also discuss completeness of analyses and measurements.

The text defines completeness as the ratio of the number of valid results obtained to the analytical results requested. This definition should be revised to express completeness as the ratio of valid results obtained to the number of samples planned for analysis.

5. Section 3.6, Page 5, Paragraph 1. The text states that rinsate blank samples will be analyzed for semivolatile organic compounds (SVOC) and metals. However, Table 3-1 shows that rinsate samples will be collected for all analytes for groundwater samples. The text also states that matrix spike/matrix spike duplicate (MS/MSD) samples are to be collected for organic analyses only, but Table 3-1 shows that MS/MSD samples are designated for all analytes for groundwater and soil samples. Finally, the text states that for inorganic analyses, an MS and laboratory duplicate (LD) will be used, but for cyanide analysis, the method (SW-846 9012) specifies the use of one MS/MSD pair. The QAPjP should be revised to address these issues and resolve all discrepancies.

6. Section 3.6, Page 6, Paragraph 3. The text states that the level of quality control (QC) effort for the field measurement of groundwater pH,

specific conductance, and temperature are provided in Attachment 1. Attachment 1 is not included in the QAPjP. This discrepancy should be addressed.

7. Table 3-1, Page 7. The table shows that six investigative groundwater samples will be collected at SWMUs F and H and AOC 5. It is unclear whether six samples will be collected from each SWMU and AOC 5 or collectively from both SWMUs and AOC 5. The QAPjP should be revised to address this issue.

8. Table 3-1, Page 8. Table 3-1 shows that eight investigative samples are to be collected from AOCs 2 and 6. However, it is not clear whether eight samples will be collected from both or each of AOCs 2 and 6. The QAPjP should be revised to address this issue.

Table 3-1 also does not show which analytical method will be used to measure spontaneous combustibility and the table shows that a MS/MSD pair will be used during the spontaneous combustibility testing. The use of an MS/MSD pair for this analysis should be explained, and the table should show the analytical method used to measure spontaneous combustibility.

Finally, Table 3-1 shows that only field measurements will be conducted for surface water. However, the QAPP does not explain the intended data usage of surface water sample field measurements. The QAPP should be revised to provide a rationale for field measurements of surface water samples.

9. Table 3-1, Page 9. The table shows that cyanide analysis will be conducted using method SW-846 8150, which is the method for analysis of herbicides. The table should be corrected to show the correct method for cyanide, which is SW-846 9012.

10. Footnote 4 to Table 3-1 states that for MS/MSDs, triple the normal sample volumes will be collected for VOC analysis. This footnote should be revised to state that three independent samples will be collected for VOCs to avoid splitting one sample into three subsamples in the laboratory. Sample splitting may result in VOC losses.

11. Footnote 6 to Table 3-1 states that groundwater samples will be analyzed for filtered and unfiltered metals during the first round and for filtered metals only during the second round. However, the QAPjP does not discuss the rounds of groundwater sampling. The QAPjP should be revised to address this issue.

12. A footnote should be added to Table 3-1 to explain what the blank cells in the table represent.

13. Tables 3-2 through 3-12. These tables report data quality objectives for SVOCs, VOCs, polychlorinated biphenyls (PCB), herbicides, organophosphorus pesticides, metals, and sulfide in water and soil or sediment samples in terms of RPD, MS/MSD, and laboratory control samples (LCS). The RPD is calculated based on the results of MS/MSDs. However, MS/MSDs and LCSs are types of samples and not a QA parameter. The QAPjP should be revised to present an



appropriate QA parameter, such as percent recovery, instead of MS/MSDs and LCSs.

14. Table 3-2 and 3-3. These tables list terphenyl-d14, fluorophenol, and phenol-d5 as surrogate compounds. However, the names of these compounds are incorrect. The correct names are p-terphenyl-d14, 2-fluorophenol, and phenol-d6. The QAPjP should be revised to correct the names of these compounds.

15. Table 3-4 and 3-5. These tables list bromofluorobenzene as a surrogate compound. However, the name of this compound is inaccurate. The correct name is 4-bromofluorobenzene. The QAPjP should be revised to correct the name of this compound.

16. Tables 3-8 and 3-9. These tables present data quality objectives for organophosphorus pesticides in water and solid samples to be analyzed by SW-846 8141. However, this analysis is not included in Table 3-1. The QAPjP should be revised to resolve this discrepancy.

17. Tables 3-10 and 3-11. These tables present data quality objectives for metals in water and soil or sediment samples in terms of RPD, MS/MSD, and LCS. However, for metals, the text states that only MSs are to be analyzed but not MSDs. The QAPjP should be revised to address this discrepancy.

Also, these tables show that thallium will be analyzed using method SW-846 7841. However, Table 7-2 shows that thallium will be analyzed using method SW-846 6010. The QAPjP should be revised to resolve this discrepancy.

Finally, Table 3-10 shows that mercury in groundwater samples will be analyzed using method SW-846 7471. However, this method is to be used to analyze mercury in solid samples. The method for water samples is SW-846 7470. Table 3-10 should be corrected accordingly.

18. Table 3-12. This table presents data quality objectives for cyanide and sulfide in water and solid samples. However, Table 3-1 indicates that no analysis for sulfide is to be conducted. The QAPjP should be revised to address this discrepancy.

19. Table 3-13, Pages 23 and 24. The title of this table indicates that it presents precision, accuracy, and completeness objectives for TCLP parameters. However, the table presents only accuracy measurements. The QAPjP should be revised to address this discrepancy.

**Section 4.0, Sampling Procedures** - This section and accompanying tables and figures contain numerous deficiencies. Examples of these deficiencies are presented below, along with some recommended revisions.

1. Section 4.1.4, Page 4, Paragraph 6. The text states that sediment samples will be collected from SWMU E to be analyzed for hazardous waste characteristics. However, Table 3-1 does not include SWMU E as one of the SWMUs to be sampled.

2. Section 4.1.4, Page 5, Paragraph 2. The text states that five samples of the spent filter cake will be collected and analyzed to assess its ability to combust spontaneously. However, the text does not explain how the number of samples to be analyzed was determined. The QAPjP should be revised to provide a rationale for the number of samples to be collected.

3. Section 4.1.4, Page 5, Paragraph 3. The text states that if groundwater gradients show that groundwater is contained in the extraction radius, no analytical testing of groundwater will be conducted. However, the text does not describe the criteria that will be used to determine if groundwater is contained in the extraction radius, thus the exclusion of analytical testing is not acceptable. The QAPjP should be revised to address this issue.

4. Section 4.1.4, Page 6, Paragraph 5. The text states that five groundwater monitoring wells are proposed in this area, and nine extraction wells currently are operating. However, the text does not explain how many wells will be sampled. The QAPjP should be revised to specify the number of wells to be sampled.

5. Section 4.1.4, Page 7, Paragraph 3. The text states that sampling in the vicinity of the Coke Plant will be limited to the chemicals listed in Table 4-1. Table 4-1 shows that one of the contaminants of interest is sulfide. However, Table 3-1 does not include sulfide as one of the analytes of interest for groundwater and soil samples. The QAPjP should be revised to address this discrepancy.

The text does not specify the number of soil samples to be collected. The text states that Figure 4-6 illustrates sampling locations for AOC 2. However, Figure 4-6 does not show soil sampling locations for AOC 2. The QAPjP should be revised to address these issues.

6. Section 4.1.4, Page 9, Paragraph 3. The text states that five monitoring wells are proposed for AOC 5 and that two monitoring wells and nine extraction wells are currently present. However, the text does not explain which wells will be sampled. The QAPP should be revised to specify the number of wells to be sampled.

7. Section 4.1.4, Page 9, Paragraph 4. The text states that soil samples that exhibit the highest vapor content as measured with a photoionization detector (PID) or organic vapor analyzer (OVA) will be analyzed for 1,2-dichloropropane (PDC) and bis(2-chloro-isopropyl)ether (BCE). However, BCE is an SVOC; therefore, the use of a PID or OVA may not reliably identify areas with high concentrations of BCE. The QAPP should be revised to address this issue.

8. Section 4.1.4, Page 10, Paragraph 3. The text states that four soil samples will be collected from AOC 7. However, the text does not provide any rationale for the number of samples to be analyzed from this AOC. The QAPjP should be revised to present this rationale.

9. Section 4.1.4, Page 11, Paragraph 2. The text states that investigatory actions planned for AOC 9 during this RFI include analyzing groundwater

samples for propylene oxide and its degradation products. However, the text does not explain which groundwater wells will be sampled and how many samples will be analyzed. Also, Table 3-1 does not include AOC 9 as one of the areas to be sampled. The QAPjP should be revised to include this information.

10. Section 4.2.2, Page 12. The text states that field blanks will be prepared by pouring distilled or deionized water through the sampling device, and then into the sample container. However, text in Section 3.6 refers to this sample as the "equipment rinsate blank." The QAPjP should be revised to consistently name this type of sample.

The text also states that distilled or deionized water will be used to collect field blanks and trip blanks. However, the quality of distilled or deionized water may be inadequate for determining blank contamination with organics. Analyte-free water such as high performance liquid chromatography (HPLC)-grade water should be used instead. The QAPjP should be revised accordingly.

11. Table 4-2 and 4-3. These tables show that cyanide and sulfide are physical properties. However, cyanide and sulfide are chemical analyses. The QAPjP should be revised to resolve this discrepancy.

12. Table 4-2, Page 18. Footnote 6 of this table states that sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) will be added to samples for analysis of base-neutral and acid extractable organics (BNA) in the presence of residual chlorine. However, the footnote does not explain how the presence of residual chlorine will be determined. Also, the table does not specify the amount of  $\text{Na}_2\text{S}_2\text{O}_3$  to be added. The footnote should be revised to provide this information.

13. Footnote 7 of Table 4-2 states that for the analysis of aldrin, the samples should be preserved by adding 0.008 percent sodium sulfate. Footnote 7 should be revised to state that for the analysis of aldrin, the samples should be preserved by adding 0.008 percent  $\text{Na}_2\text{S}_2\text{O}_3$ .

**Section 5.0, Custody Procedures** - This section has several deficiencies. Examples of these deficiencies are presented below, along with recommended revisions.

1. Section 5.1, Pages 1 and 2. This section does not discuss the following items: (1) field logbooks, (2) sample tags, (3) sample shipping cooler custody seals, (4) the sample numbering system, and (5) sample shipping procedures. The QAPjP should be revised to provide this information.

2. Section 5.2, Page 2. This section does not identify the laboratory sample custodian. Also, the text does not present specific procedures for logging in the samples, tracking samples internally, storing samples, and disposing of samples and sample extracts, and digestates. The QAPjP should be revised to address these issues.

3. Section 5.3, Pages 2 and 3. The text states that the RFI consultant project manager will be the custodian of all the project files not in the possession of the project coordinator. However, the QAPjP does not specify

the period of time the final evidence file will be maintained before disposal. The QAPP should be revised to address this issue. The QAPjP should also state that the final evidence file must be offered to U.S. EPA prior to disposal.

**Section 6.0, Calibration Procedures and Frequency** - This section and accompanying table have several deficiencies. Examples of these deficiencies are presented below, along with some recommended revisions.

1. Section 6.1, Pages 1 and 2. This section does not discuss the calibration of other instruments to be used in the field such as the turbidity meter, redox potential meter, and dissolved oxygen meter. The QAPjP should be revised to discuss the calibration procedures of all instruments used in the field.

2. Section 6.2, Pages 2 through 4. This section presents generic information for the Enseco-North Canton laboratory. The QAPjP should be revised to discuss only project-specific information.

3. Table 6-1, Pages 5 through 8. In some cases, this table presents information for calibration procedures as they relate to analytes that will be determined without presenting the instruments that will be used for these analyses. In other cases, the table presents information for the calibration of certain instruments without specifying the analytes that will be determined by these instruments. For example, in one case, the table presents calibration procedures for pesticides and PCBs without specifying the instrument that will be calibrated. In another case, the table presents calibration procedures for the ion-specific electrode and spectrophotometer without specifying which analytes will be determined by these instruments. Also, the table discusses calibration procedures for "traacs 800," but it is not clear what this term refers to. Finally, the table discusses the calibration of "miscellaneous inorganic analysis," that is vague and therefore, unacceptable. Table 6-1 should be revised in accordance with Table 6 of the Model QAPjP to present calibration procedures for the determination of each analyte of interest and for each instrument that will be used to determine these analytes. Also, extraneous information should be removed from this table, and unclear terms should be clarified. For each instrument, the table should present calibration acceptance criteria and corrective actions to be taken when these criteria are not met.

**Section 7.0, Analytical and Measurement Procedures** - This section and accompanying tables present several deficiencies. Examples of these deficiencies are presented below, along with recommended revisions.

1. Tables 7-3 through 7-9. These tables summarize detection limits for the analytes of interest in an inconsistent manner. For example, Tables 7-3 through 7-8 summarize method detection limits (MDL) and practical quantitation limits (PQL) for groundwater and solid samples. Table 7-9 presents detection limits for TCLP metals, pesticides, herbicides, VOCs, and SVOCs without qualifying if these detection limits are MDLs or PQLs. Because both MDLs and PQLs are presented, it is unclear what the project-specific target reporting

limits (TRL) are. Tables 7-3 through 7-9 should be revised to specify TRLs for all contaminants of concern.

In addition, for several compounds, the MDLs are higher than the target levels presented in the Michigan Department of Natural Resources (MDNR) Act 307 type B cleanup criteria. Examples include but are not limited to the following:

Table 7-3 shows that the MDLs for 1,2,4,5-tetrachlorobenzene, pentachlorobenzene, and pentachlorophenol in groundwater samples are 6.72, 4.27, and 2.36 micrograms per liter ( $\mu\text{g/L}$ ). However, the MDNR Act 307 type B acceptable MDLs for these compounds in the same order are 0.1, 0.5, and 1  $\mu\text{g/L}$ , respectively. Also, Table 7-3 shows that the MDL for 1,2,4,5-tetrachlorobenzene in soil samples is 224 micrograms per kilogram ( $\mu\text{g/kg}$ ). However, the MDNR Act 307 type B acceptable MDL for this compound is 20  $\mu\text{g/Kg}$ .

Table 7-4 shows that the MDLs for chloromethane; ethylbenzene; methylene chloride; toluene; and 1,2-dichloropropane in groundwater samples are 1.26, 1.32, 2.41, 1.51, and 1.23  $\mu\text{g/L}$ . However, the MDNR Act 307 type B acceptable MDL for these compounds is 1  $\mu\text{g/L}$ . Therefore, alternative analytical methods may need to be used in order to obtain acceptable target reporting limits.

Table 7-4 also presents identical MDLs and PQLs for soil and water samples, which is unlikely because of the different matrices involved. The revised QAPjP should address this issue.

Table 7-8 does not present an MDL for sulfide in soil samples.

Tables 7-3 through 7-9 should be revised to present the project-specific TRLs and the MDNR Act 307 type B acceptable target MDLs.

**Section 8.0, Internal Quality Control Checks** - The content of this section is generic. This section should be revised to provide project-specific information.

### **Section 9.0, Data Reduction, Validation, and Reporting**

The content of this section is generic. This section should be revised to provide project-specific information.

**Section 10.0, Performance and Systems Audits** - This section and accompanying tables and figure contain several deficiencies. Examples of these deficiencies are presented below, along with some recommended revisions.

1. Section 10.0, Page 1, Paragraph 2. The text states that Figure 10-1 shows the audit flow chart. However, this figure is not included in the QAPjP. The QAPjP should be revised to include this figure.

The text also states that the QAO and the project manager will be responsible for identifying and performing audits. However, the names of the QAO and the

project manager, as well as their affiliation(s), have not been identified. The QAPP should be revised to provide this information.

2. Section 10.2.1, Pages 2 and 3. The information presented in this section is generic. This section should be revised to present project-specific information.

**Section 11.0, Preventative Maintenance** - This section and accompanying table contain several deficiencies. Examples of these deficiencies are presented below, along with recommended revisions.

1. Section 11.2, Pages 1 and 2. The information presented in this section is generic. This section should be revised to present project-specific information.

2. Table 11-1, Pages 3 through 7. This table includes generic information that is not directly relevant to the project-specific analytes. This table should be revised to describe preventive maintenance procedures only for instrumentation related to the project-specific analytes.

Also, according to its title, Table 11-1 should present preventive maintenance procedures only for laboratory equipment. However, preventive maintenance for field equipment, such as the specific conductivity meter, dissolved oxygen meter, and turbidity meter, is also discussed. Table 11-1 should be revised to include only relevant information. Also, the QAPjP should be revised to include a table that summarizes preventive maintenance procedures for field equipment.

**Section 12.0, Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness** - This section presents equations for calculating accuracy and precision based on LCSs and MS/MSDs. However, for certain parameters, such as pH and specific conductivity, accuracy and precision are not to be determined by analyzing MS/MSDs. This section should be revised to include equations for calculating accuracy and precision for all analytes to be determined.

**Section 13.0, Corrective Action** - The content of this section is generic. This section should be revised to provide project-specific information. This section should also discuss the involvement of U.S. EPA in implementing corrective actions.

**Section 14.0, Quality Assurance Reports to Management** - The content of this section is generic. This section should be revised to provide project-specific information.

## II. OTHER

### A. GENERAL COMMENTS

Section 3004(v) of RCRA, as amended by the Hazardous and Solid Waste Amendments of 1984 (HSWA), authorizes the U.S. EPA to require corrective action beyond the facility boundary where appropriate. In the U.S. EPA's discussions with BASF, starting with negotiations of the draft §3008 (h) Corrective Action Order, Detroit River sediments have been and continue to be identified as a remediation, human health and ecological risk concern.

As summarized in the AOC, the U.S. EPA's and the Michigan Department of Natural Resources (MDNR) records, as well as the records of BASF (e.g., SSP&A, 1984) clearly indicate that prior to the installation of the current groundwater pump-and-treat system at the North Works, groundwater from the BASF North Works flowed to the Detroit River. Demonstration of the effectiveness of the current groundwater pump-and-treat system at the BASF North Works in removing contamination from all sources continues to be the major corrective action objective. However, the effectiveness of the treatment system has not yet been established. Even if the U.S. EPA agreed that the pump-and-treat system is the most effective remediation measure of contaminated groundwater, it does not, and cannot, solely demonstrate that historical releases to the Detroit River adjacent to the BASF North Works have been assessed, and if necessary and feasible, addressed.

As discussed at the October 17, 1994, meeting, the RFI serves to focus the scope of the corrective action, and is tailored to the specific conditions and circumstances of a facility. In addition, the U.S. EPA endeavors to minimize unnecessary and unproductive investigations. Accordingly, the U.S. EPA is willing to allow BASF to develop a schedule to phase assessment and investigation of Detroit River sediment contamination into the RFI, but the total absence of any sediment assessment and investigation in the RFI Workplan is unacceptable. BASF must revise the RFI Workplan to address the assessment and investigation of contaminated sediments in the Detroit River adjacent to the North Works facility.

The approach to be followed in assessing and investigation contamination of Detroit River sediments must be developed by BASF. However, an acceptable approach, that addresses the U.S. EPA's primary concerns, would resemble the following general outline:

## ON SITE INVESTIGATION

Sampling and analysis of sewers and sumps, and SWMUs/AOCs (e.g., Coke Plant), proximal to the Detroit River to identify hazardous wastes and constituents of concern (e.g., LNAPLS).

Assessment of past and present potential for groundwater transport to Detroit River and contamination of near shore sediments. Including unconsolidated soils and fill that were subject to erosion prior to installation of metal sheet piling. Identify possible "tracer" (e.g., free-phase oil, PAHs, etc.) that may be used for assessment and confirmation of past and/or present groundwater transport.

Assessment of current corrective measure, i.e., pump-and-treat system and sheet piling installation. If not effective source(s) control, identify additional corrective measures.

## BATHYMETRY STUDY OF NEAR SHORE

Bathymetry study to determine boundaries of previous dredging and sediment channel interface(s). Desk-top review and/or field data collected on adjacent Detroit River characteristics (e.g., depth, flow, velocity, transport, deposition, substrate, sediment characterization, etc.)

Develop sampling plan, or document no need for action.

Sediment Sampling and Analysis.

Corrective Measure Study and Implementation, including assessment of remediation feasibility if up-stream contamination continues, etc.



In conclusion, the RFI Workplan in its entirety fails to address Detroit River sediments. Revisions to the RFI Workplan are necessary that will allow the development of an acceptable approach to sediment assessment, investigation and remediation. Comments on the QAPjP and associated Plans identify some of the areas where sediments must be addressed, but the burden is on BASF to prepare and revise the RFI Workplan accordingly.

## B. DESCRIPTION OF CURRENT CONDITIONS

The Description of Current Conditions (DCC) includes no information in the characteristics of environmental setting regarding the Detroit River, and possible contamination of sediments. The DCC should be revised to include background information on what is known about Detroit River sediments in the Detroit Rivers, especially adjacent to the North Works. Such information should include contamination, deposition, benthos, transport, bathymetry, substrate characteristics, dredging dates and analysis, etc. U.S. Army Corps of Engineers, Michigan Department of Natural Resources and other pertinent references should be noted, such as the Report provided to the U.S. EPA in BASF's November 21, 1994, letter, and the examples noted below.

More specifically, examples of revisions that should be made, include the following:

- Pertains to QAPjP*
- 1.1.1* Section 1.3, Bullet *2* of QAPjP - Include release from the North Works;
  - of QAPjP* 2. Section 1.1, Objective 6 - An objective must be added on addressing ecological risks, that includes the United States Fish and Wildlife Services identification of two Federal endangered/threatened species in the Detroit River.
  3. Section 11.6 - As stated in the QAPjP, a justification must be made for these sampling depths.
  4. Section 12.6 - See Comment 3 above, and documentation, if available (e.g., aerial photography series), should be presented that this area was not a landfill.
  5. Section 13.6 - Analysis should include sediments in the Detroit River.
  6. Section 15 - Groundwater analysis must be more clearly defined and justified.
  7. Section 18.6 - Groundwater analysis will be required regardless of whether contamination continues to leave facility.
  8. Section 19.6 - Please include more specifics.

## **C. PRE-INVESTIGATION EVALUATION OF CORRECTIVE MEASURE TECHNOLOGIES**

Remediation of contaminated sediments in the Detroit River must be included in the Evaluation, including an assessment of any known limitations of sediment remediation based on characteristics of the Detroit River, ecological concerns, cost concerns, etc.

## **D. RFI WORKPLAN**

### **1. PROJECT MANAGEMENT PLAN**

DQOs must be extensively revised as evident by preceding comments. assessment and investigation of contaminated sediments in the Detroit River must be listed as a project objective, as well as any necessary subsequent tasks. As discussed at the October 17, 1994, meeting it may be appropriate to develop a phased approach, e.g., delay sampling until it can be determined whether what contaminants are present in groundwater, and whether contamination continues to flow to the Detroit River. However, such a approach should not be used to avoid developing and specifying sampling network design, target parameters, SOPs and analytical methods, etc., as long as acknowledgement is made that subsequent revisions may be necessary and are subject to the approval of the U.S. EPA.

### **2. DATA MANAGEMENT PLAN**

Detroit River sediments data must be included.

### **3. HEALTH AND SAFETY PLAN**

Contaminated sediment assessment, investigation (sampling and analysis) considerations must be added.

### **4. PUBLIC PARTICIPATION PLAN**

Participation in the RAP and BPAC workgroups should be detailed.

## **REFERENCES**

October 1994 - Detroit River AOC Contaminated Sediments - Draft Report of the Technical Workgroup

December 1988 - Integrated Study of Exposure and Biological Effects of In-Place Sediment Pollutants in the Detroit River: An Upper Great Lakes Connecting Channel

SIGNATURE/INITIAL CONCURRENCE REQUESTED - RCRA ENFORCEMENT BRANCH (REB)								
SC/BR/OFC SECRETARY								
INITIATOR /AUTHOR	IL/IN TES CHIEF	MI/WI TES CHIEF	MN/OH TES CHIEF	IL/MI/WI EPS CHIEF	IN/MN/OH EPS CHIEF	REB BRANCH CHIEF	RCRA ASSOC. DIR.	WMD DIVISION DIRECTOR
<i>[Signature]</i> 11/7/95								

*had*  
1/25/95

**BASF Corporation**

**BASF**

December 21, 1994

**CERTIFIED MAIL  
RETURN RECEIPT REQUESTED  
Z 100 492 164**

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (HRE-8J)  
77 West Jackson Street  
Chicago, Illinois 60604

Subject: Verification of telephone conversation on 12/21/94

Dear Ms. Sharrow:

This is to confirm our telephone conversation on December 21, 1994. During the October 17 meeting, EPA stated that the RFI consultant had to be specified in the final Work Plans. I asked you if this was an EPA Policy. I said that BASF would not be able to pick a consultant and revise the plans within 60 days. I also stated that we would not be able to obtain an accurate cost estimate for the work because the work plans will be significantly revised. You stated that you would investigate to determine if specifying the RFI consultant is an EPA requirement and send a written response by the end of next week.

Concerning EPA's final comments on the work plans, you said that the comments should be issued in early January.

You requested two changes on the distribution of the monthly progress report and I agreed. Mr. P. Schrantz (MDNR Lansing) will be replaced with Ms. Rhonda Blayer who is on his staff. The second change is that Mr. Larry Aubuchon (MDNR Livonia) will be added to the distribution.

If this verification does not accurately reflect our conversation, please advise.

Thank you.

Sincerely yours,

*Bruce Roberts*

Bruce Roberts  
Project Coordinator

**RECEIVED**  
JAN 01 1995  
**OFFICE OF RCRA  
WASTE MANAGEMENT DIVISION  
EPA REGION V**

PLAN FOR SELECTING THE RFI CONSULTANT  
AT THE BASF NORTH WORKS FACILITY

The BASF plan for selecting the RFI Consultant for the RCRA Corrective Action at the North Works facility is a multi step process. In summary, the major steps are (1) request bids from several consultants, (2) evaluate bids, (3) select top three consultants, (4) request revised RFP, (5) evaluate bids and select the potential RFI consultant, and (6) obtain a contract with the consultant.

Below is complete listing of the steps in the process with actual dates for completed activities and target dates for remaining activities.

1. Request statement of qualifications from several consultants - December 1994
2. Evaluate statement of qualifications and select the top six consultants - December 1994
3. Issue RFP based upon June 1994 Work Plans (bids were due January 31) - January 12, 1995
4. Evaluate the RFPs and select the top three consultants - February 21, 1995  
(The bids were evaluated independently by the BASF team members. Selection criteria considered were corporate qualifications, project organization, qualifications of assigned team, technical approach, support services, and cost estimate.)
5. Joint meeting with the top three consultants to discuss the facility - March 14, 1995
6. Visit by BASF team members at the top consultants' office to learn more about the consultant - week of March 27, 1995
7. Issue revised RFP based upon March 1995 Work Plans - by April 5, 1995 (RFP would be due by April 19, 1995)
8. Evaluate revised RFPs and select the potential top consultant - week of May 1, 1995
9. Obtain contract with top consultant - week of May 22, 1995
10. Notify EPA - week of May 29, 1995

11 August 1994

HRE-8J

Ms. Rhonda Blayer  
State of Michigan  
Department of Natural Resources  
Waste Management Division  
Post Office Box 30028  
Lansing, Michigan 48909

-and-

Ms. Mary Vanderleen  
State of Michigan  
Department of Natural Resources  
SouthEast Michigan District Office  
38980 Seven Mile Road  
Livonia, Michigan 481532

RE: RCRA Corrective Action  
BASF Corporation  
North Works  
Wyandotte, Michigan  
MID 064 197 742

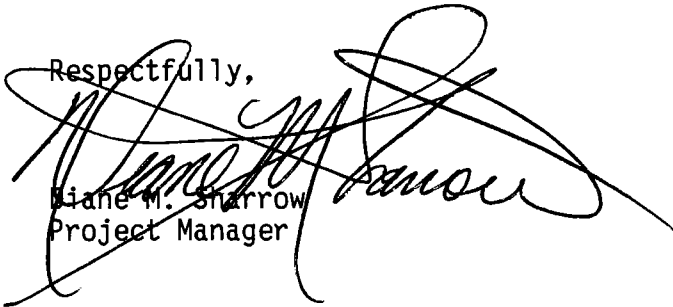
Dear Ms. Blayer and Ms. Vanderleen:

Enclosed you will find a copy of the RCRA Facility Investigation (RFI) Workplans, minus the Quality Assurance Project Plan, submitted to the United States Environmental Protection Agency (U.S. EPA), Region 5, under a RCRA §3008 Administrative Order on Consent, Docket No. V-W-011-94. A copy of the Quality Assurance Project Plan (QAPjP) will be forwarded to you by BASF Corporation.

Although U.S. EPA is the implementing Agency with regards to this Order, your comments are being solicited. U.S. EPA would like to ensure that any Michigan Act 64 or Act 307 concerns regarding the North Works site be addressed as early on the RCRA corrective action process as possible. We are asking that you pay particular attention to the utilization of Michigan Type B cleanup criteria (§4 of the QAPjP).

If you have any questions regarding this request please contact me at (312) 886-6199. The U.S. EPA would like to begin compiling all comments on the RFI Workplans no later than September 30, 1994. Thank you for your assistance.

Respectfully,

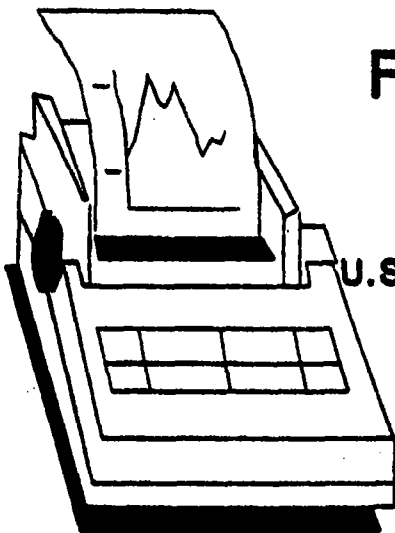


Diane M. Sharrow  
Project Manager

Enclosures

cc: Adam Bickel, BASF Corporation

FACSIMILE SENT  
BY msedek DATE 8/15  
3:30



## FACSIMILE REQUEST

RCRA ENFORCEMENT BRANCH  
U.S. ENVIRONMENTAL PROTECTION AGENCY  
REGION 5

77 WEST JACKSON (HRE-8J)  
CHICAGO, ILLINOIS 60604

TO: Adam Bickel  
OFFICE/PHONE (313) 246-5142  
FACSIMILE NUMBER (313) 246-6774  
VERIFICATION NUMBER \_\_\_\_\_

---

FROM: Diane Sharrow  
OFFICE/PHONE (312) 886-6199  
DATE 08/15/94 NUMBER OF PAGES 3

Additional comments:

SENT FROM FACSIMILE NUMBER  
(312) 353-4788



**BASF Corporation**

Certified Mail (P 267 573 603)  
Return Receipt Requested

**BASF**

May 27, 1994  
4E07014-400

Ms. Diane Sharrow  
Project Manager  
United States Environmental Protection Agency  
Region V, (HRE-8J)  
77 West Jackson Street  
Chicago, Illinois 60604

Subject: Submittal of Work Plans  
RCRA Facility Investigation  
Docket No.: V-W-011-94  
BASF Corporation, Wyandotte, Michigan

Dear Ms. Sharrow:

BASF Corporation submits three copies of the RFI work plans for the Wyandotte facility.

The Plans are found in the following order:

- Project Management Plan;
- Data Management Plan;
- Health and Safety Plan;
- Public Participation Plan; and
- Pre-Investigation Evaluation of Corrective Measure Technologies.

As agreed, the Quality Assurance Project Plan and Current Conditions Report will be submitted to EPA by June 27, 1994.

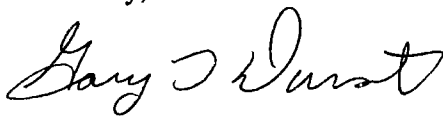
I certify that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to evaluate the information submitted. I certify that the information contained in or accompanying this submittal is true, accurate, and complete. As to those identified portion(s) of this submittal for which I cannot personally verify the accuracy, I certify that this submittal and all attachments were prepared in accordance with procedures designed to assure that qualified personnel properly gathered and

DET21472

Ms. Diane Sharrow  
United States Environmental Protection Agency  
May 27, 1994  
Page 2

evaluated the information submitted. Based on my inquiry of the person or persons who manage the system, or those directly responsible for gathering the information, or the immediate supervisor of such person(s), the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

Sincerely,



Gary T. Durst  
Wyandotte Site Manager

JCL:kan

Enclosures

cc: A. Bickel - BASF  
J.C. Lanigan, Jr. - WCC

DET21472

May 19, 1994

Ms. Diane Sharrow  
U.S. EPA Region 5  
RCRA Enforcement Branch, HRE-8J  
77 West Jackson Boulevard  
Chicago, IL 60604-3590

**RECEIVED**  
MAY 25 1994

**OFFICE OF RCRA**  
**WASTE MANAGEMENT DIVISION**  
**EPA, REGION V**

RE: MID 064197742 - Request for 30 Day Extension

Dear Ms. Sharrow:

Attachment II of the Consent Order requires submission of the RFI reports and workplans (i.e., Tasks I, II, and III) within 90 days of the effective date of the Order. Taking into consideration the holiday, the reports and workplans are due to EPA by May 31, 1994.

During our telephone conversation on May 17, 1994, the issue of a extension was discussed and EPA recommended that BASF submit a formal request for such an extension. In keeping with that recommendation, BASF Corporation formally requests a 30 day extension to the deadline of May 31, 1994 for the Current Conditions Report and the Quality Assurance Project Plan.

BASF will submit five out of the seven reports and workplans by May 31, 1994. The five reports and workplans will include the Pre-Investigation Evaluation of Corrective Measure Technologies Report, Project Management Plan, Data Management Plan, Health and Safety Plan, and the Public Participation Plan. We hope that the EPA will recognize this as a good faith demonstration of BASF's commitment toward fulfilling our obligations.

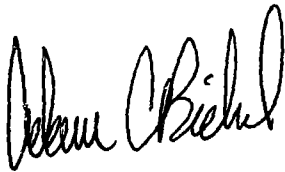
BASF's reasons for requesting an extension include the complex historical nature of the site. Obtaining the information necessary for the completion of the AOC/SWMU and facility background sections of the Current Conditions Report was complicated by inaccessibility of key individuals and documents. The effort required more time than anticipated to incorporate as much historical information as possible. The extra time necessary to obtain this information, effectively narrowed the internal timeline for which the consultant was allotted to compile and edit the information for the Current Conditions Report.

The information obtained from the Current Conditions Report must be considered to develop an appropriate Quality Assurance Project Plan. Therefore, BASF also requests the extension to apply to the Quality Assurance Project Plan.

Request for 30 Day Extension  
May 19, 1994  
Page 2

BASF believes that the time required of all parties for the review and approval of the RFI reports and workplans will be best served with the submission of complete and higher quality documents. If you have any questions, please call me at (313) 246-5142.


Sincerely,



Adam C. Bickel  
Corporate Ecology and Safety

acb

cc: G. Durst - BASF  
J. Lanigan - WCC

Fold at line over top of envelope to the right of the return address	<b>CERTIFIED</b> P 267 573 602 <b>MAIL</b>	P. 267 573 602	
		 <b>Receipt for Certified Mail</b>	
		No Insurance Coverage Provided Do not use for International Mail (See Reverse)	
		Sent to <b>Ms. Diane Sharrow</b> <b>U.S. EPA Region 5</b>	
		Street and No. <b>77 West Jackson Blvd.</b>	
		P.O., State and ZIP Code <b>Chicago, IL 60604-3590</b>	
		Postage	\$ <b>.39</b>
		Certified Fee	<b>1.00</b>
		Special Delivery Fee	
		Restricted Delivery Fee	
Return Receipt Showing to Whom & Date Delivered			
Return Receipt Showing to Whom, Date, and Addressee's Address	<b>1.35</b>		
TOTAL Postage & Fees	\$ <b>2.74</b>		
Postmark or Date			
BASF Corporation 1600 Liddle Ave. Wyandotte, MI 48192 Attn: Tox Dept. (A. Bickel)			

PS Form 3800, June 1991

**CONTRACT NUMBER: 68-W4-000-**

**WA #: R050-**

**STATEMENT OF WORK for  
RFI, CMS, CMI REVIEW  
BASF - NORTH WORKS  
WYANDOTTE, MICHIGAN**

**A. BACKGROUND**

The BASF, Inc., (BASF), site is located at 1609 Biddle Ave., in Wyandotte, Michigan, and totals approximately 230 acres, with its eastern border formed by the Detroit River/Trenton Channel. While under the ownership of BASF, the facility known as the North Works, has engaged in the manufacture and conducted research and pilot activities in support of manufacturing, industrial inorganic chemicals, polyether polyol resins, etc. Historical activities date to the late 1800's, and include the manufacture of soda ash and coke.

Approximately  $\frac{1}{2}$  to  $\frac{2}{3}$ 's of the facility is reclaimed marshland and riverbottom, filled to bring the site to the approximate present grade with cinders, limestone, gravel, cobble, coal, timbers, concrete, etc. The fill material occupies the full length of the facility in a wedge 22 feet thick near Biddle Ave., extending to 1000 feet wide to the north, to about 2400 feet in width across the center of the facility. Surficial fill, fluvial sand and peat make up the upper-most hydrogeologic system at the facility, with undetermined hydraulic communication with hydrogeologic systems beyond the facility boundary.

Current on-site activity includes a preparation of a RCRA Facility Investigation (RFI). Technical review of the RFI, with an emphasis on the hydrogeological evaluation of the current pump and treat system, is required to determine whether contamination is reaching the Detroit River.

**B. ENFORCEMENT ACTION**

U.S. EPA and BASF entered into a Consent Order under Section 3008(h) of RCRA on February 14, 1994. This Consent Order requires BASF to conduct an RFI, a Corrective Measures Study (CMS), and Corrective Measures Implementation (CMI), if necessary.

**C. DESCRIPTION OF TASKS TO BE PERFORMED BY THE CONTRACTOR**

- (1) Prepare workplan based upon statement of work and submit the workplan to U.S. EPA for review and approval.
- (2) Provide technical support to U.S. EPA as the On-Site Project Coordinator during the facility's performance of any CMS/CMI. This will include documenting and inspecting some work performed and recommending to U.S. EPA that BASF perform additional work or that BASF stop work.
- (3) Review of RFI, CMS Workplan, and CMS and any necessary revisions submitted by BASF as required by the Consent Order. \*

\* Reviews should focus on the complex hydrogeology at the facility and be based upon relevant U.S. EPA guidance, technical adequacy and current scientific thinking. Additionally, reviews must focus on making sure scientific rationales are presented by BASF so proper documentation is presented in the deliverable.

- (4) As directed by U.S. EPA WAM, the contractor may conduct community relations activities such as: (1) assisting EPA in preparing the decision documents and fact sheets for release to the public, (2) providing public meeting support such as logistical support for one public meeting including scheduling and announcing the public meeting, preparing press releases, making technical presentations, obtaining the services of a qualified and reputable court reporter\*, preparing graphics or visual aids, (3) assisting EPA in the preparation and placement of newspaper ads to inform the local community of upcoming public meetings and formal public comment periods, (4) creating and maintaining a mailing list, and
- (5) making arrangements for, creating and maintaining public information repository(ies), on a ongoing basis.

**D. DELIVERABLES REQUIRED AND SCHEDULE FOR COMPLETION OF TASKS**

- (1) [Task 1]. Contractor shall provide a workplan within twenty (20) days after acknowledgment of receipt.

- (2) [Task 2 through 3 above]. Provide U.S. EPA with briefings, as requested, by telephone or in person, at a mutually agreed upon time, on the progress of the RFI and CMS reviews or onsite work evaluated. Topics covered during these briefings should include progress of the work to be performed, or any additional information relevant to the corrective action process.
- (3) [Tasks 2 through 3 above]. Provide U.S. EPA with a detailed review of the RFI and CMS Reports submitted by BASF. Contractor shall review documents and provide written comments to U.S. EPA within twenty-five (25) days of receipt.
- (4) [Tasks 2 through 3 above]. Perform field oversight activities, as directed by U.S. EPA, to assure that BASF is abiding by approved plans and procedures. Maintain a log of BASF's field activities and a photographic log of major site activities. Provide U.S. EPA with written status reports documenting field activities. The report must include progress of work, problems encountered, deviations from approved procedures and plans. Contractor shall provide field activity reports within seven (7) days of completion of field oversight activity.
- (5) [Tasks 2 through 3 above]. Review other documents submitted by BASF to support the corrective action process. Contractor shall review documents and provide written comments to U.S. EPA within twenty-five (25) days of receipt.
- (6) [Tasks 4 through 5 above]. Except for ongoing activities, within 20 days of request by WAM, but transcripts are due two weeks after meeting.

**E. GENERAL**

Upon issuance of written technical direction, the contractor shall submit for inspection copies of all work in progress at any time under this work assignment.

The contractor shall develop and maintain files supporting each work assignment.

**F. PRINTING RESTRICTIONS**

The contractor is prohibited from performing any printing under the Government Printing and binding Regulations. Duplication is allowed to the extent it does not exceed the 5,000 impressions of a single-page document or 25,000 impressions of a multiple-page document and the work cannot be performed under the job or time constraints at the EPA Print Shop. If the total number of photocopies for this work assignment exceeds 5,000 copies the contractor shall identify the photocopying costs by task and deliverable.

**G. WORK ASSIGNMENT CONFLICT OF INTEREST CERTIFICATION**

The Contractor shall provide a conflict of interest certification in the work plan. In the certification, the Contractor must certify that, to the best of the Contractor's knowledge and belief, all actual or potential organizational conflicts of interest have been reported to the Contracting Officer or that, to the best of the Contractor's knowledge and belief, no actual or potential organizational conflicts of interest exist. In addition, the Contractor must certify that its personnel who perform work under this work assignment or relating to this work assignment have been informed of their obligation to report personal and organizational conflicts of interest to the Contractor recognizes its continuing obligation to identify and report any actual or potential conflicts of interest arising during performance of this work assignment or other work related to this work assignment.



CONTRACT #: 68-W4-000-  
WA #: R050--

5

#### **H. PERIOD OF PERFORMANCE**

The period of performance for this work assignment is from date of Contracting Officer approval through the date specified in Block 22 of the REPA Work Assignment Form (WAF).

#### **I. TRAVEL**

The Contractor is required to follow the requirement of subpart 31.2 of the FAR and the Federal regulations in incurring allowable travel costs under this work assignment, and correspondingly must at all times seek and obtain government rates whenever available and observe current subsistence ceilings.

#### **J. LEVEL OF EFFORT**

The Contractor shall not exceed the estimated LOW or dollar amount in Block 20 of the Work Assignment Form.

In addition to the notification requirements of FAR Clauses 52.232-20 and FAR 52.232-22 in this contract, the Contractor shall notify the CO and Project Officer in writing when 75% and 80% respectively of the LOE and/or dollars for this WA have been expended. If applicable, the notice shall state the estimated amount of addition funds required to continue performance of the WA and the reasons why additional funds are necessary. The notice shall also state the estimated date when authorized LOE and/or funding will be depleted.

#### **K. INFORMATION COLLECTION**

If it becomes a contractual requirement to collect identical information from ten (1) or more public respondents, the Paperwork Reduction Act of 1980, 44 U.S. C 3501 applies. In that event, the contractor shall not take any action to solicit information from any of the public respondents until notified in writing by the Contracting Officer that the required Office of Management and Budget (OMB) final clearance has been received. A public respondent is defined as any person or organization which is not a U.S. Federal Government agency or an employee therefore, State employees and their employees are therefore

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classified as public respondents.

Soliciting identical information applies to any collection method, i.e., written, oral, electronic, etc.

Any question of applicability of the PRA shall be resolved by submitting a complete description of the circumstances in a written request to the Contracting Officer. No collection shall be undertaken until the Contracting Officer provides written notice to the contractor as to the applicability of the PRA. If the PRA is determined to be applicable, the Contractor shall not initiate any collection until the requisite approval is received.

#### **L. TECHNICAL DIRECTION**

The Work Assignment Manager (WAM) is authorized to provide technical direction which clarifies the state of work as set forth in this work assignment. Other than the designated WAS, only the Project Officer and Contracting Officer are authorized to give technical direction.

Technical direction will be issued in writing or confirmed in writing, by the WAM, within five (5) calendar days after verbal issuance. The WAM will forward a copy of the technical direction memorandum to the Contracting Officer and a copy to the Project Officer. If the Contractor has not received written confirmation within 5 calendar days of verbal issuance, the contractor must so notify the Project Officer and Contracting Officer.

Technical direction must be within the contract and the work assignment statement of work.

Technical direction includes (1) direction to the contractor which assists the contractor in accomplishing the Statement of Work and (2) comments on and approval of reports and other deliverables.

The Contracting Officer is the only person authorized to make changes to this work assignment or contract. Any changes must be approved by the Contracting Officer in writing, as an amendment to this work assignment and/or a modification to the contract.

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**M. CONFIDENTIAL BUSINESS INFORMATION**

If this work assignment requires use of RCRA Confidential Business Information (CBI), the contractor shall abide by all RCRA CBI requirements, the stipulations found in the RCRA CBI Security Manual and the CBI clauses in the contract.

**N. CONTRACTOR IDENTIFICATION**

To avoid any perception that contractor personnel are EPA employees, the contractor shall assure that contractor personnel are clearly identified as independent contractors of EPA when attending meetings with outside parties or visiting field sites.